REMARKS

Entry of the foregoing Preliminary Amendment prior to examination of this application is respectfully requested.

The present application claims priority from and is a U.S. national application of PCT/GB00/02584 filed July 5, 2000. Claims 3, 8, 12, 15, 17, 19, 21-28, 30, 32-37, 40, 42 and 44-46 have been amended. Claim 47 has been cancelled without prejudice or disclaimer. Claims 1-46 are now pending. The claims are amended to change the claim dependencies.

Attached hereto is a marked-up version of the changes made to the application by this Preliminary Amendment.

Respectfully submitted,

Date: 1/15/02

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Reg. No.: P-50,732

Telephone No.: 216-241-6700
Facsimile No.: 216-241-8151

Attachment: Version with Markings to Show Changes Made

VERSION WITH MARKINGS TO SHOW CHANGES MADE

In the Claims:

A paragraph has been added after the title and before the first paragraph in the application. The purpose of this paragraph is to claim priority to the corresponding PCT application.

The following claims have been amended as follows:

3. (Amended) A method according to [claims 1 or 2] claim 1, characterised in that the subsequent di-hydrate is formed into pellets.

8. (Amended) A method according to [any of claims 5 to 7] claim 5, characterised in that the calcining is hydrothermal to form α calcium sulphate hemi-hydrate.

12. (Amended) A method according to [any of claims 5 to 7] claim 5, characterised in that the calcining is carried out in dry heat conditions.

15. (Amended) A method according to [any of claims 8 to 14] claim 8, characterised in that the calcining is carried out for a period of a half to six hours.

17. (Amended) A method according to [any of claims 5 to 15] claim 5, characterised in that following calcining, the calcium sulphate hemi-hydrate is ground to a powder.

19. (Amended) A method according to [any of the previous claims] claim 1, characterised in that the initial calcium sulphate di-hydrate is formed by mixing soluble calcium and sulphate salts such that calcium sulphate precipitates out.

21. (Amended) A method according to [claims 19 or 20] claim 19, characterised in that the calcium salt is a chloride.

22. (Amended) A method according to [claims 19 or 20] claim 19, characterised in that the calcium salt is a nitrate.

23. (Amended) A method according to [any of claims 19 to 22] claim 19, characterised in that the sulphate is a sodium salt.

24. (Amended) A method according to [any of claims 19 to 22] claim 19, characterised in that the sulphate is a potassium salt.

25. (Amended) A method according to [any of claims 19 to 22] claim 19, characterised in that the sulphate is [a] an ammonium salt.

26. (Amended) A method according to [any of claims 19 to 25] claim 19, characterised in that the calcium and sulphate salts are provided in a substantially equal molecular ratio.

27. (Amended) A method according to [any of claims 1 to 18] claim 1, characterised in that the initial calcium sulphate di-hydrate is formed from neutralising lime with sulphuric acid.

28. (Amended) A method according to [any of the previous claims] claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate takes place within a temperature range 110-350°C.

30. (Amended) A method according to [any of claims 1 to 27] claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate takes place at a temperature above 350°C to form insoluble anhydrite.

32. (Amended) A method according to [any of the preceding claims] claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate by the application of heat takes place in an open container.

33. (Amended) A method according to [any of claims 1 to 31] claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate by the application of heat takes place in a closed container.

34. (Amended) A method according to [any of the preceding claims] claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate by the application of heat takes place hydrothermally in the presence of steam.

35. (Amended) A method according to [any of the preceding claims] claim 1, characterised in that the rehydration of the calcium sulphate anhydrite takes place immediately following dehydration.

36. (Amended) A method according to [any of the preceding claims] claim 1, characterised in that the calcium sulphate anhydrite is fully immersed in water for rehydration.

37. (Amended) A method according to [any of claims 1 to 35] claim 1, characterised in that the calcium sulphate anhydrite is fully immersed in a dilute salt solution for rehydration.

40. (Amended) A method according to [any of claims 37 to 39] claim 37, characterised in that the concentration of the salt solution is less than 1%.

42. (Amended) A method according to [any of the previous claims] claim 1, characterised in that finely powdered calcium sulphate di-hydrate is added to be present during rehydration such that the powdered calcium sulphate acts as crystal seeds.

44. (Amended) A method according to [claims 28 or 29, or any of claims 32 to 43 when dependent on claims 28 or 29] claim 28, characterised in that the rehydration takes less than five days.

45. (Amended) A method according to [any of the preceding claims] claim 1, characterised in that the subsequent calcium sulphate di-hydrate is dried following crystallisation.

46. (Amended) A method according to claim 8 [or any of claims 9 to 44 when dependent on claim 8], characterised in that the subsequent calcium sulphate di-hydrate is held in a damp condition prior to calcining.

Claim 47 has been cancelled without prejudice or disclaimer.

S.N. 10/031501

CLAIM STATUS

1. (Currently Amended) A method of producing surgical grade calcium sulphate characterised in that the method comprises forming an initial calcium sulphate di-hydrate from synthetic constituents; dehydrating the initial calcium sulphate di-hydrate to form calcium sulphate anhydrite; and subsequently rehydrating the calcium sulphate anhydrite by immersing in an aqueous environment and allowing subsequent calcium sulphate di-hydrate to crystallise out.

2. (Original) A method according to claim 1, characterised in that the subsequent di-hydrate is used as a solid material bone filler.

3. (Previously Amended) A method according to claim 1, characterised in that the subsequent di-hydrate is formed into pellets.

4. (Original) A method according to claim 3, characterised in that the crystallized subsequent calcium sulphate di-hydrate is ground prior to forming into pellets.

5. (Original) A method according to claim 1, characterised in that the subsequent calcium sulphate di-hydrate is calcined to form calcium sulphate hemi-hydrate.

6. (Original) A method according to claim 5, characterised in that the calcium sulphate hemi-hydrate is mixed with water to form a settable paste.

7. (Original) A method according to claim 5, characterised in that the calcium sulphate hemi-hydrate is mixed with a salt solution to form a settable paste.

8. (Previously Amended) A method according to claim 5, characterised in that the calcining is hydrothermal to form α calcium sulphate hemi-hydrate.

9. (Original) A method according to claim 8, characterised in that the calcining is carried out in an autoclave.

10. (Original) A method according to claim 9, characterised in that the calcining is carried out at a pressure of 1-6 bar.

11. (Original) A method according to claim 10, characterised in that the calcining is carried out at 2-3 bar.

12. (Previously Amended) A method according to claim 5, characterised in that the calcining is carried out in dry heat conditions.

13. (Original) A method according to claim 12, characterised in that the calcining is carried out at a temperature of 70-200° C.

14. (Original) A method according to claim 13, characterised in that the calcining is carried out at 150-175° C.

15. (Previously Amended) A method according to claim 8, characterised in that the calcining is carried out for a period of a half to six hours.

16. (Original) A method according to claim 15, characterised in that the calcining is carried out for one to two hours.

17. (Previously Amended) A method according to claim 5, characterised in that following calcining, the calcium sulphate hemi-hydrate is ground to a powder.

18. (Original) A method according to claim 17, characterised in that the powder has particle size of less than 150 microns.

19. (Previously Amended) A method according to claim 1, characterised in that the initial calcium sulphate di-hydrate is formed by mixing soluble calcium and sulphate salts such that calcium sulphate precipitates out.

20. (Original) A method according to claim 19, characterised in that the initial di-hydrate thus formed is washed, and subsequently filtered, crushed and/or dried.

21. (Previously Amended) A method according to claim 19, characterised in that the calcium salt is a chloride.

22. (Previously Amended) A method according to claim 19, characterised in that the calcium salt is a nitrate.

23. (Previously Amended) A method according to claim 19, characterised in that the sulphate is a sodium salt.

24. (Previously Amended) A method according to claim 19, characterised in that the sulphate is a potassium salt.

25. (Previously Amended) A method according to claim 19, characterised in that the sulphate is an ammonium salt.

26. (Previously Amended) A method according to claim 19, characterised in that the calcium and sulphate salts are provided in a substantially equal molecular ratio.

27. (Previously Amended) A method according to claim 1, characterised in that the initial calcium sulphate di-hydrate is formed from neutralising lime with sulphuric acid.

28. (Previously Amended) A method according to claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate takes place within a temperature range 110-350°C.

29. (Original) A method according to claim 28, characterised in that the dehydration of the initial calcium sulphate di-hydrate takes place at less than 300°C.

30. (Previously Amended) A method according to claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate takes place at a temperature above 350°C to form insoluble anhydrite.

31. (Original) A method according to claim 30, characterised in that the rehydration takes more than five days.

32. (Previously Amended) A method according to claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate by the application of heat takes place in an open container.

33. (Previously Amended) A method according to claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate by the application of heat takes place in a closed container.

34. (Previously Amended) A method according to claim 1, characterised in that the dehydration of the initial calcium sulphate di-hydrate by the application of heat takes place hydrothermally in the presence of steam.

35. (Previously Amended) A method according to claim 1, characterised in that the rehydration of the calcium sulphate anhydrite takes place immediately following dehydration.

36. (Previously Amended) A method according to claim 1, characterised in that the calcium sulphate anhydrite is fully immersed in water for rehydration.

37. (Previously Amended) A method according to claim 1, characterised in that the calcium sulphate anhydrite is fully immersed in a dilute salt solution for rehydration.

38. (Original) A method according to claim 37, characterised in that the salt solution comprises succinic acid.

39. (Original) A method according to claim 37, characterised in that the salt solution comprises potassium sulphate solution.

40. (Previously Amended) A method according to claim 37, characterised in that the concentration of the salt solution is less than 1%.

41. (Original) A method according to claim 40, characterised in that the concentration of the salt solution is substantially 0.1%.

42. (Previously Amended) A method according to claim 1, characterised in that finely powdered calcium sulphate di-hydrate is added to be present during rehydration such that the powdered calcium sulphate acts as crystal seeds.

43. (Original) A method according to claim 42, characterised in that addition is in the order of 5g per litre of water.

44. (Previously Amended) A method according to claim 28, characterised in that the rehydration takes less than five days.

45. (Previously Amended) A method according to claim 1, characterised in that the subsequent calcium sulphate di-hydrate is dried following crystallisation.

46. (Previously Amended) A method according to claim 8, characterised in that the subsequent calcium sulphate di-hydrate is held in a damp condition prior to calcining.

47. (Cancelled)

48. (New) A method according to claim 1 wherein the aqueous environment comprises a salt.

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=> file reg
FILE 'REGISTRY'
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=> display history full l1-


     FILE 'REGISTRY'
                      E CALCIUM SULFATE/CN
L1              1 SEA "CALCIUM SULFATE"/CN

     FILE 'HCA'
L2          35859 SEA L1 OR (CALCIUM# OR CA)(W)SULFATE# OR CASO4
L3         148754 SEA BONE OR BONES
L4        1038712 SEA DEHYDRAT? OR DRY? OR DRIED OR DESSICA? OR DESSICCA?
                  OR DESICCA? OR DESICA? OR LYOPHIL? OR ANHYDROUS? OR
                  ANHYDRAT?
L5         126739 SEA DEHYDRAT?
L6         200355 SEA HYDRAT? OR REHYDRAT?
L7                QUE CRYST? OR RECRYST?
L8          86446 SEA CALCIN?
L9          19627 SEA DI(W)HYDRAT? OR DIHYDRAT?

     FILE 'REGISTRY'
                  E CALCIUM CHLORIDE/CN
L10             1 SEA "CALCIUM CHLORIDE"/CN
                  E CALCIUM NITRATE/CN
L11             1 SEA "CALCIUM NITRATE"/CN
                  E SODIUM SULFATE/CN
L12             1 SEA "SODIUM SULFATE"/CN
                  E POTASSIUM SULFATE/CN
L13             1 SEA "POTASSIUM SULFATE"/CN
                  E AMMONIUM SULFATE/CN
L14             1 SEA "AMMONIUM SULFATE"/CN
                  E LIME/CN
                  E CALCIUM OXIDE/CN
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                  E SULFURIC ACID/CN
L16             1 SEA "SULFURIC ACID"/CN

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L18        401378 SEA L16 OR SULFURIC#(A)ACID# OR H2SO4
L19          4946 SEA L17 AND L18
L20            34 SEA L19 AND L3
L21             3 SEA L20 AND L9
L22            13 SEA L20 AND L2
L23            15 SEA L20 AND L4
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L24          0 SEA L20 AND L5
L25          3 SEA L20 AND L6
L26         12 SEA L20 AND L7
L27          1 SEA L20 AND L8
L28          6 SEA L22 AND L23
L29          5 SEA L22 AND L26
L30          7 SEA L23 AND L26
L31      84087 SEA L10 OR CACL2
L32      13332 SEA L11 OR CA(W)NO3(W)2
L33      74425 SEA L12 OR NA2SO4
L34      22953 SEA L13 OR K2SO4
L35      22185 SEA L14 OR NH4(W)2(W)SO4
L36       6739 SEA (L31 OR L32) AND (L33 OR L34 OR L35)
L37         28 SEA L36 AND L3
L38          2 SEA L37 AND L2
L39         11 SEA L37 AND L4
L40          0 SEA L37 AND L5
L41          3 SEA L37 AND L6
L42          3 SEA L37 AND L7
L43          0 SEA L37 AND L8
L44          1 SEA L37 AND L9

     FILE 'REGISTRY'
               E CALCIUM SULFATE DIHYDRATE/CN
L45          1 SEA "CALCIUM SULFATE DIHYDRATE"/CN

     FILE 'HCA'
L46         21 SEA (INITIAL? OR NASCEN? OR NEWBORN? OR NEW)(3A)(L45 OR
               CASO4(2A)(2H2O OR 2(A)H2O) OR (CALCIUM# OR CA)(3A)SULFATE
               #(3A)(DI(A)HYDRATE# OR DIHYDRATE#))
L47          1 SEA L46 AND L3

     FILE 'REGISTRY'
L48          1 SEA 10101-41-4
L49          1 SEA 14798-04-0

     FILE 'HCA'
L50       1227 SEA L48
L51       4722 SEA L49
L52         44 SEA L50 AND L51
L53          1 SEA L52 AND L3
L54          1 SEA L37 AND L20
L55       5180 SEA L45 OR CASO4(2A)(2H2O OR 2(A)H2O) OR (CALCIUM# OR
               CA)(3A)SULFATE#(3A)(DI(A)HYDRATE# OR DIHYDRATE#)
L56         46 SEA L55 AND L3
L57      13473 SEA L1
L58         14 SEA L56 AND L57
L59          6 SEA L46 AND L4
L60          6 SEA L46 AND L5
L61         10 SEA L46 AND L6
L62         13 SEA L46 AND L7
L63          0 SEA L46 AND L8
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L64             0 SEA L46 AND L19
L65             7 SEA L61 AND L62
L66             1 SEA L46 AND L17
L67             3 SEA L46 AND L18
L68             1 SEA L46 AND (L31 OR L32)
L69             4 SEA L46 AND (L33 OR L34 OR L35)
L70            37 SEA L21 OR L25 OR L27 OR L28 OR L29 OR L30 OR L38 OR L41
                  OR L42 OR L44 OR L47 OR L53 OR L54 OR L59 OR L60 OR L65
                  OR L66 OR L67 OR L68 OR L69
L71            23 SEA (L39 OR L58 OR L61 OR L62) NOT L70
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L70 ANSWER 1 OF 37 HCA COPYRIGHT 2003 ACS on STN

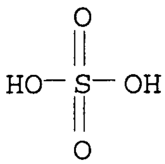
138:406993 Bioactive ceramic coating for artificial **bone**. Li, Panjian (Depuy Orthopaedics, Inc., USA). U.S. US 6569489 B1 20030527, 19 pp., Cont.-in-part of U.S. Ser. No. 38,444. (English). CODEN: USXXAM. APPLICATION: US 2000-574358 20000519. PRIORITY: US 1998-38444 19980311.

AB A bioactive **bone** mineral carbonated nano-**cryst**. apatite is chem. bonded to a variety of substrates, including implantable prostheses. This coating is applied uniformly to substrate surfaces of varying geometry and surface textures. It is firmly secured to the substrate and encourages rapid and effective **bone** ingrowth. The coating is applied by immersing the substrate in an aq. soln. contg. calcium, phosphate and carbonate ions. Other ions, such as sodium, potassium, magnesium, chloride, sulfate, and silicate, may optionally be present in the soln. The soln. is exposed in a controlled environment when it reacts with the substrate to form the coating. A synthetic **bone** apatite mineral film formed on the sand-blasted surface of the Ti6Al4V disks, was heated at 120, 200, 300, 400, 450, 500, 550, 600, 650.degree. and then analyzed by FTIR. The FT-IR spectroscopy indicated that synthetic apatite **bone** mineral contained chem. absorbed water in its structure and not hydroxyl ions.

IT **7757-82-6**, Sodium sulfate, processes **10043-52-4**, Calcium chloride, processes
(bioactive ceramic coating and method)

RN 7757-82-6 HCA

CN Sulfuric acid disodium salt (8CI, 9CI) (CA INDEX NAME)



2 Na

RN 10043-52-4 HCA
CN Calcium chloride (CaCl2) (9CI) (CA INDEX NAME)

Cl— Ca— Cl

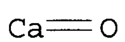
IC ICM A61L027-00
ICS A61K027-00; B05D003-12
NCL 427002260; 427002240; 427002270; 427435000; 427443200
CC 63-7 (Pharmaceuticals)
ST artificial **bone** ceramic coating
IT **Bone**
(artificial; bioactive ceramic coating and method)
IT 3812-32-6, Carbonate, processes 7440-70-2, Calcium, processes 7447-40-7, Potassium chloride (KCl), processes 7647-14-5, Sodium chloride, processes **7757-82-6**, Sodium sulfate, processes 7758-11-4 7786-30-3, Magnesium chloride (MgCl2), processes **10043-52-4**, Calcium chloride, processes 12627-13-3, Silicate 14066-19-4, Hydrogen phosphate, processes 14265-44-2, Phosphate, processes 14808-79-8, Sulfate, processes 16887-00-6, Chloride, processes
(bioactive ceramic coating and method)

L70 ANSWER 2 OF 37 HCA COPYRIGHT 2003 ACS on STN
136:150666 Raw materials of Venetian glassmakers as recorded in formulation books from the 14th century to the first half of the 20th century. Part II. List of primary and secondary raw materials and semifinished products. Moretti, Cesare (S. Vito Al Tagliamento, Pordenone, Italy). Rivista della Stazione Sperimentale del Vetro (Murano, Italy), 31(3), 17-32 (Italian) 2001. CODEN: RSSVDT. ISSN: 0391-4259. Publisher: Stazione Sperimentale del Vetro.
AB In 1999, research was carried out on numerous Muranese glassmaker recipe manuscripts; raw materials were classified with a brief commentary on the basis of their function in the batch and/or in the glass structure. Most of these unpublished recipe books were discovered in private collections a few years ago. In this second part of the report, a full list is reported of primary and secondary raw materials, as well as intermediate products, as recorded in different recipe books from the 14th to 20th centuries. For each

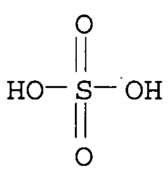
term, given mostly in the original lexicon use, the nature, chem. formula, and function in the recipe are briefly indicated, with addnl. historical information on the material, and a mention, when possible, of relevant supply sources in the past.

IT **1305-78-8**, Calcium monoxide, properties **7664-93-9**, **Sulfuric acid**, properties
(raw materials for Venetian glass as recorded in manuscripts from 14th to 20th centuries)

RN 1305-78-8 HCA
CN Calcium oxide (CaO) (9CI) (CA INDEX NAME)



RN 7664-93-9 HCA
CN Sulfuric acid (8CI, 9CI) (CA INDEX NAME)



CC 20-3 (History, Education, and Documentation)
Section cross-reference(s): 57

IT Charcoal
(**bone**; raw materials for Venetian glass as recorded in manuscripts from 14th to 20th centuries)

IT 64-19-7, Acetic acid, properties 301-04-2, Salt of saturn 471-34-1, Calcium carbonate, properties 497-19-8, Soda ash, properties 584-08-7, Potassium carbonate 868-14-4, Cream of tartar 1302-83-6, Lazurite 1303-33-9, Arsenic trisulfide 1303-96-4, Sodium tetraborate decahydrate 1305-62-0, Calcium hydroxide, properties **1305-78-8**, Calcium monoxide, properties 1309-37-1, Ferric oxide, properties 1309-38-2, Magnetite, properties 1309-64-4, Antimony trioxide, properties 1313-13-9, Manganese dioxide, properties 1314-13-2, Zinc monoxide, properties 1314-41-6, Red lead oxide 1317-36-8, Lead monoxide, properties 1317-39-1, Dicopper monoxide, properties 1317-60-8, Hematite, properties 1317-63-1, Limonite 1317-66-4, Marcasite 1319-45-5, Azurite 1319-46-6, Basic lead carbonate 1327-53-3, Arsenic trioxide 1333-82-0, Chromium trioxide 1345-04-6, Antimony trisulfide 6080-56-4 7439-89-6, Iron, properties 7439-92-1, Lead, properties 7439-96-5, Manganese, properties 7439-97-6, Mercury, properties 7440-02-0, Nickel, properties 7440-22-4, Silver, properties 7440-31-5, Tin, properties 7440-43-9, Cadmium, properties 7440-50-8, Copper, properties 7440-57-5, Gold, properties 7487-94-7, Mercury dichloride, properties 7631-99-4, Sodium nitrate, properties 7647-14-5, Sodium chloride, properties **7664-93-9**, **Sulfuric acid**, properties 7697-37-2, Nitric acid, properties

7704-34-9, Sulfur, properties 7720-78-7, Iron vitriol 7733-02-0, Zinc sulfate 7757-79-1, Potassium nitrate, properties 7757-82-6, Sodium sulfate, properties 7758-98-7, Copper sulfate, properties 7778-50-9, Potassium bichromate 7782-49-2, Selenium, properties 7783-90-6, Silver chloride, properties 7803-52-3, Stibine 8007-56-5, Aqua regia 9000-01-5, Gum arabic 10025-91-9, Antimony trichloride 10043-67-1, Aluminum potassium sulfate 10101-41-4, Calcium sulfate **dihydrate** 11104-61-3, Cobalt oxide 11113-93-2, Uranium oxide 12044-30-3, Realgar 12070-39-2, Copper carbonate hydroxide (Cu3(CO3)2(OH)2) 12125-02-9, Sal ammoniac, properties 12143-43-0, Lead-tin yellow 12255-89-9, Orpiment 12597-69-2, Steel, properties 12668-36-9 12777-38-7, Arsenic oxide 13462-86-7, Barite 14476-15-4, Cerussite 14476-25-6, Calamine 14567-86-3, Chrysocolla 14635-80-4, Kalinite 14639-89-5, Chalcedony 14808-60-7, Quartz, properties 15096-52-3, Cryolite 15491-24-4, Natron 16389-88-1, Dolomite, properties 18282-10-5, Tin dioxide 19122-79-3, Cinnabar 52036-90-5, Sulfur fluoride 56320-22-0, Arsenic disulfide 56729-51-2, Arsenic tetrasulfide 58471-11-7, Ferrugine 74891-45-5, Massicot 79497-69-1, **Sulfuric acid**
, iron potassium salt
(raw materials for Venetian glass as recorded in manuscripts from 14th to 20th centuries)

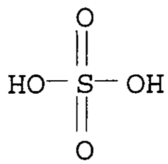
L70 ANSWER 3 OF 37 HCA COPYRIGHT 2003 ACS on STN
134:118030 Method for manufacture of surgical grade calcium sulfate. Cooper, John Joseph (Biocomposites Limited, UK). PCT Int. Appl. WO 2001005706 A1 20010125, 17 pp. DESIGNATED STATES: W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, BF, BJ, CF, CG, CH, CI, CM, CY, DE, DK, ES, FI, FR, GA, GB, GR, IE, IT, LU, MC, ML, MR, NE, NL, PT, SE, SN, TD, TG. (English). CODEN: PIXXD2. APPLICATION: WO 2000-GB2584 20000705. PRIORITY: GB 1999-16601 19990716.

AB A method of producing surgical grade calcium sulfate suitable for use as resorbable osteoconductive **bone** void filler material. The method comprises forming an **initial calcium sulfate dihydrate** from synthetic constituents, **dehydrating** the **initial calcium sulfate dihydrate** to form **calcium sulfate** anhydrite, and subsequently **rehydrating** the calcium sulfate anhydrite and allowing subsequent calcium sulfate dihydrate to **crystallize** .

IT **10101-41-4P**, Calcium sulfate dihydrate
(method for manuf. of surgical grade calcium sulfate)

RN 10101-41-4 HCA

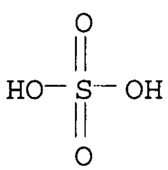
CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



Ca

2 H_2O

IT 14798-04-0, Anhydrite (Ca(SO4))
(method for manuf. of surgical grade calcium sulfate)
RN 14798-04-0 HCA
CN Anhydrite (Ca(SO4)) (9CI) (CA INDEX NAME)



Ca

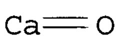
IC ICM C01F011-46
ICS A61L027-02
CC 49-5 (Industrial Inorganic Chemicals)
Section cross-reference(s): 63
ST surgical grade calcium sulfate manuf; resorbable osteoconductive **bone** void filler calcium sulfate manuf
IT **10101-41-4P**, Calcium sulfate dihydrate
(method for manuf. of surgical grade calcium sulfate)
IT **14798-04-0**, Anhydrite (Ca(SO4))
(method for manuf. of surgical grade calcium sulfate)

L70 ANSWER 4 OF 37 HCA COPYRIGHT 2003 ACS on STN
133:155507 Implant comprising calcium cement and hydrophobic liquid. Bohner, Marc (Mathys Robert Stiftung, Switz.; Stratec Medical A.-G.). PCT Int. Appl. WO 2000045867 A1 20000810, 40 pp. DESIGNATED STATES: W: AU, CA, CN, JP, KR, US; RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE. (English). CODEN: PIXXD2. APPLICATION: WO 1999-EP684 19990202.

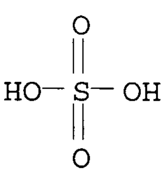
AB The compn. comprises a hydraulic cement for implantation in the human or animal body, said hydraulic cement comprising a first component comprising a calcium source and a second component comprising water, which hardens after mixing of the components. The compn. further comprises a third component with a hydrophobic liq. The compn. allows to obtain a cement with open macroporosity enabling a rapid **bone** ingrowth. A mixt. of .alpha.-tri-calcium phosphate 8, pptd. tricalcium phosphate 0.8, calcium cement 0.5 g, Cremophor EL 0.001, and Tegosoft M 8.0 mL were stirred for 4 min. The mixt. was then poured into a syringe and injected into a cavity. After hardening, the cavity was filled with an open macroporous calcium phosphate structure.

IT **1305-78-8**, Calcium oxide, biological studies
7664-93-9, **Sulfuric acid**, biological studies
(implant comprising calcium cement and hydrophobic liq.)

RN 1305-78-8 HCA
CN Calcium oxide (CaO) (9CI) (CA INDEX NAME)



RN 7664-93-9 HCA
CN Sulfuric acid (8CI, 9CI) (CA INDEX NAME)



IC ICM A61L024-02
ICS A61L027-02; A61L027-12
CC 63-7 (Pharmaceuticals)
IT 57-10-3, Palmitic acid, biological studies 57-11-4, Stearic acid, biological studies 59-02-9, .alpha.-Tocopherol 60-33-3, 9,12-Octadecadienoic acid (9Z,12Z)-, biological studies 77-92-9, biological studies 84-66-2, Diethylphthalate 100-51-6, Benzenemethanol, biological studies 102-71-6, biological studies 102-76-1, Triacetin 105-54-4, Ethylbutyrate 106-32-1, Ethyl caprylate 106-33-2, Ethyl laurate 107-92-6, Butyric acid, biological studies 109-43-3, Di butyl sebacate 110-27-0, Isopropyl myristate 110-38-3, Ethyl caprate 111-61-5, Ethyl stearate 111-62-6, Ethyl oleate 112-80-1, Oleic acid, biological studies 112-86-7, Erucic acid 119-13-1, .delta.-Tocopherol 120-51-4, Benzyl benzoate 121-54-0, Benzethonium chloride 123-66-0, Ethyl caproate 124-06-1, Ethyl myristate 124-07-2, Caprylic acid, biological studies 126-44-3, Citrate ion, biological studies 141-22-0, Ricinoleic acid 141-43-5, biological studies 142-62-1, Hexanoic acid, biological studies 142-91-6, Isopropyl palmitate 143-07-7, Lauric acid, biological

studies 148-03-8, .beta.-Tocopherol 151-21-3, Sodium lauryl sulfate, biological studies 334-48-5, Capric acid 373-49-9, Palmitoleic acid 463-40-1, Linolenic acid 471-34-1, Calcium carbonate, biological studies 506-30-9, Arachidic acid 544-35-4, Ethyl linoleate 544-63-8, Myristic acid, biological studies 577-11-7, Docusate sodium 620-17-7 628-97-7, Ethyl palmitate 1191-41-9, Ethyl linolenate 1305-62-0, Calciumhydroxide, biological studies **1305-78-8**, Calcium oxide, biological studies 1306-01-0, Tetracalcium phosphate 1306-05-4, Fluorapatite (Ca5F(PO4)3) 1306-06-5, Hydroxyapatite 1306-07-6, Calcium oxide phosphate (Ca10O(PO4)6) 1338-39-2, Sorbitan monolaurate 1338-41-6, Sorbitan monostearate 1338-43-8, Sorbitan monooleate 7616-22-0, .gamma. Tocopherol 7664-38-2, Phosphoric acid, biological studies **7664-93-9**, **Sulfuric acid**, biological studies 7757-93-9, Dicalcium phosphate 7758-23-8, Monocalcium phosphate 7758-87-4, .alpha.-Tricalcium phosphate 7789-77-7, Dicalcium phosphate **dihydrate** 8007-43-0, Sorbitan sesquioleate 8014-38-8, Emulsifying wax BP 8044-71-1, Cetrimide 9000-65-1, Tragacanth gum 9002-89-5, Polyvinyl alcohol 9002-92-0, Polyethylene glycol monolauryl ether 9003-01-4, Polyacrylic acid 9004-61-9, Hyaluronic acid 9004-62-0, Hydroxyethyl cellulose 9004-64-2, Hydroxypropyl cellulose 9004-65-3, Hydroxypropyl methyl cellulose 9004-67-5, Methyl cellulose 9004-95-9, Polyethylene glycol monocetyl ether 9004-98-2, Polyethylene glycol monooleyl ether 9004-99-3, Polyoxyl 40 stearate 9005-00-9, Polyethylene glycol monostearyl ether 9005-37-2, Propylene glycol alginate 9005-38-3, Sodium alginate 9005-64-5, Polysorbate 20 9005-65-6, Polysorbate 80 9005-66-7, Polysorbate 40 9005-67-8, Polysorbate 60 9005-70-3, Polysorbate 85 9005-71-4, Polysorbate 65 9012-76-4, Chitosan 9050-31-1, Hydroxypropyl methyl cellulose phthalate 10031-30-8, Monocalcium phosphate monohydrate 10034-76-1, Calcium sulfate hemihydrate 10086-45-0, Calcium pyrophosphate 10103-46-5, Calcium phosphate 11138-66-2, Xanthan gum 13767-12-9, Octacalcium phosphate 14000-31-8, Diphosphate 14265-44-2, Phosphate, biological studies 18281-05-5, Ethyl arachidate 22537-22-0, Magnesium ion, biological studies 25496-72-4, Glyceryl monooleate 26266-57-9, Sorbitan monopalmitate 26266-58-0, Sorbitan trioleate 26658-19-5, Sorbitan tristearate 29116-98-1, Sorbitan dioleate 51938-44-4, Sorbitansesquistearate 54392-26-6, Sorbitan monoisostearate 54392-27-7, Sorbitan triisostearate 68238-87-9, Sorbitan diisostearate 71812-38-9, Sorbitan sesqui isostearate 154362-61-5, Polysorbate120
(implant comprising calcium cement and hydrophobic liq.)

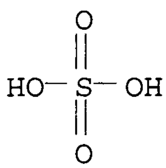
L70 ANSWER 5 OF 37 HCA COPYRIGHT 2003 ACS on STN
132:24425 Comprehensive utilization of waste liquid from regeneration of spent ion exchange resins: compound flocculant preparation. Li, Yansheng; Wen, Yang; Cao, Kui; Xu, Shifen (Dalian Railway Institute, Peop. Rep. China). Dalian Tiedao Xueyuan Xuebao, 19(2), 37-40 (Chinese) 1998. CODEN: DTXBEI. ISSN: 1000-1670. Publisher: Dalian Tiedao Xueyuan Xuebao Bianjibu.

AB The waste liq. from regenerating ion exchange resin was regarded as a raw material in manuf. of compd. coagulant. The compd. coagulant contains 8-10 % alumina.

IT **7757-82-6P**, Sodium sulfate, preparation **10101-41-4P**, **Calcium sulfate dihydrate**
(**new** product; comprehensive utilization of waste liq. from regeneration of spent ion exchange resins: compd. flocculant prepn.)

RN 7757-82-6 HCA

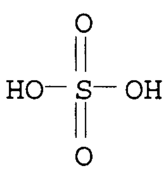
CN Sulfuric acid disodium salt (8CI, 9CI) (CA INDEX NAME)



2 Na

RN 10101-41-4 HCA

CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



Ca

2 H_2O

CC 49-3 (Industrial Inorganic Chemicals)
Section cross-reference(s): 60, 61

IT 7647-01-0P, Hydrochloric acid, preparation **7757-82-6P**, Sodium sulfate, preparation **10101-41-4P**, **Calcium sulfate dihydrate**
(**new** product; comprehensive utilization of waste liq. from regeneration of spent ion exchange resins: compd. flocculant prepn.)

L70 ANSWER 6 OF 37 HCA COPYRIGHT 2003 ACS on STN
130:236766 Method for producing feed calcium by using low usage of acid and **CaO** and defluorination. Yan, Minglang (Peop. Rep. China). Faming Zhuanli Shenqing Gongkai Shuomingshu CN 1170517 A 19980121, 7 pp. (Chinese). CODEN: CNXXEV. APPLICATION: CN 1996-110550 19960713.

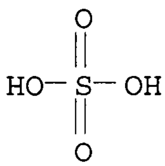
AB The method comprises (1) reaction of **H2SO4** and ground phosphate rock to prep. H3PO4 (SiO2 contained inorg. matter is added in the system during the process and reacted with F in the ground phosphate rock to form H2SiF6 and SiF4, which decreases the corrosion of HF to the prodn. facility); (2) defluorination by adding K salt (or Na salt) in the H3PO4 and reacting at 20-50.degree. for 0.5-2 h to obtain K2SiF6 (or Na2SiF6) ppt. which is sepd., **dried**, and formed K2SiF6 (or Na2SiF6) product (60-80% F in H3PO4 is recovered) and acid (HCl, **H2SO4**, and H3PO4, the total content of the acid is 35 kg/t feed Ca); (3) adding Ca3(PO4)2 salt in the defluorized H3PO4 (Ca3(PO4)2 reacts with **H2SO4**, H3PO4, and **K2SO4** (or **Na2SO4**) at 50-80.degree. for 0.5-3 h and forms Ca(H2PO4)2 and **CaSO4**); and (4) deeply defluorination at 30-60.degree. for 0.5-3 h by adding Ca2+ (such as CaCO3, **CaCl2**, **Ca(NO3)2**, Ca(OH)2, and **CaO**) and alk. in the system to pH 2.2-2.8 and P-F ratio .gtoreq.100. The SiO2 contained inorg. matter is water glass, silica, and diatomite. The K salt (or Na salt) is selected from **K2SO4**, **Na2SO4**, KCl, KNO3, K2HPO4, NaHSO4, NaH2PO4, NaCl, KOH, NaOH, K2CO3, and Na2CO3, its usage is 100-300% that of theor. defluorination. It reduces the use of **sulfuric acid** and calcium oxide, and high in purity. The addn. of Ca3(PO4)2 salt (selected from **bone** powder, Ca3(PO4)2, and CaPF6) is 70-135 kg/t H3PO4. The alk. is selected from NaOH, NH4OH, Na2CO3, KOH, and K2CO3. The addn. of Ca2+ (in deeply defluorination process) is 120-180% theor. defluorination usage. The P2O5 content in the feed Ca is 85-95%.

IT **1305-78-8**, Calcia, reactions **7664-93-9**, **Sulfuric acid**, reactions **7778-18-9**, **Calcium sulfate** **7778-80-5**, Potassium sulfate, reactions **10043-52-4**, Calcium chloride, reactions **10124-37-5**, Calcium nitrate
(method for producing feed calcium by using low usage of acid and **CaO** and defluorination)

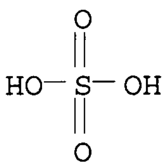
RN 1305-78-8 HCA
CN Calcium oxide (CaO) (9CI) (CA INDEX NAME)

Ca=O

RN 7664-93-9 HCA
CN Sulfuric acid (8CI, 9CI) (CA INDEX NAME)

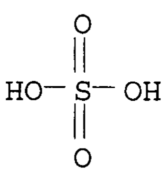


RN 7778-18-9 HCA
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

RN 7778-80-5 HCA
CN Sulfuric acid dipotassium salt (8CI, 9CI) (CA INDEX NAME)

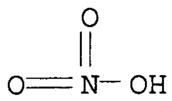


2 K

RN 10043-52-4 HCA
CN Calcium chloride (CaCl2) (9CI) (CA INDEX NAME)

Cl— Ca— Cl

RN 10124-37-5 HCA
CN Nitric acid, calcium salt (8CI, 9CI) (CA INDEX NAME)



1/2 Ca

IC ICM A23K001-175
ICS C01B033-20; C05D003-00
CC 17-12 (Food and Feed Chemistry)
IT Feed
(method for producing feed calcium by using low usage of acid and **CaO** and defluorination)
IT 7757-93-9P, Calcium hydrogen phosphate
(method for producing feed calcium by using low usage of acid and **CaO** and defluorination)
IT 471-34-1, Calcium carbonate, reactions 584-08-7, Potassium carbonate 1305-62-0, Calcium hydroxide, reactions **1305-78-8**, Calcia, reactions 1310-58-3, Potassium hydroxide, reactions 1310-73-2, Sodium hydroxide, reactions 1314-56-3, Phosphorus oxide, reactions 7447-40-7, Potassium chloride, reactions 7601-54-9, Sodium phosphate 7631-86-9, Silica, reactions 7631-99-4, Sodium nitrate, reactions 7647-01-0, Hydrochloric acid, reactions 7647-14-5, Sodium chloride, reactions 7664-38-2, Phosphoric acid, reactions 7664-39-3, Hydrogen fluoride, reactions 7664-41-7, Ammonia, reactions **7664-93-9**, **Sulfuric acid**, reactions 7681-38-1, Sodium bisulfate 7757-79-1, Potassium nitrate, reactions **7778-18-9**, **Calcium sulfate** 7778-53-2, Potassium phosphate **7778-80-5**, Potassium sulfate, reactions 7789-74-4, Calcium fluorophosphate **10043-52-4**, Calcium chloride, reactions 10103-46-5, Calcium phosphate **10124-37-5**, Calcium nitrate 16871-90-2, Potassium fluosilicate 16893-85-9, Sodium fluosilicate 16961-83-4, Fluosilicic acid 39384-00-4, Silicon fluoride
(method for producing feed calcium by using low usage of acid and **CaO** and defluorination)

L70 ANSWER 7 OF 37 HCA COPYRIGHT 2003 ACS on STN
129:71090 Effects of gypsum dihydrate on **hydration** of the 3CaO.Al2O3-CaSO4.2H2O-CaCO3 system. Lee, Jong-Kyu; Ohba, Yoko; Sakai, Etsuo; Daimon, Masaki (Fac. Eng., Tokyo Inst. Technol., Tokyo, 152, Japan). Muki Materiaru, 5(274), 194-199 (Japanese) 1998. CODEN: MUMAFX. ISSN: 1340-7899. Publisher: Sekko Sekkai Gakkai.
AB The **hydration** mechanism of 3CaO.cntdot.Al2O3(C3A)-CaSO4.cntdot.2H2O-CaCO3 system and the effects of the amt. of CaSO4.cntdot.2H2O were discussed based on the XRD quant. anal., and

the possibility of Delayed Ettringite Formation was also discussed. In the case of C3A-**CaSO4**.cntdot.**2H2O** system, the **initial hydration** of C3A was delayed by the formation of gel contg. of SO42-. The early **hydration** of C3A was further delayed by addn. of CaSO4.cntdot.2H2O and CaCO3 because the gel contg. SO42- and CO32- was produced on the surface of C3A. When the reaction of CaSO4.cntdot.2H2O was finished, the reaction of CaCO2 was started in the case of C3A-CaSO4.cntdot.2H2O-CaCO3 system. Delayed ettringite formation would take place because monosulfoaluminate is not stable in the presence of CaCO3. In the presence of CaCO3, the SO42- groups in the interlayer region of monosulfoaluminate are replaced by CO32-. This reaction leads to the formation of monocarboaluminate, and to increasing the sulfate concn. in the soln., and hence to the **recrystn**. of ettringite. To prevent the delayed ettringite formation, the redn. of monoulfoaluminate formation is important. The delayed ettringite formation occurred in the C3A-2/3CaSO4.cntdot.2H2O-15 mass% CaCO3 system. When the mole ratio of CaSO4.cntdot.2H2O/C3A is 1/3, the delayed ettringite formation did not occur until 28 days. But a large amt. of monosulfoaluminate is produced and unreacted 3CaO.cntdot.Al2O3 and CaCO3 remain. Therefore, delayed ettringite formation will be occur in the long term. In the C3A-CaSO4.cntdot.2H2O-15 mass% CaCO3 system, delayed ettringite formation is not obsd. Therefore, by increasing the amt. of CaSO4.cntdot.2H2O addn., the delayed ettringite formation can be prevented.

CC 58-1 (Cement, Concrete, and Related Building Materials)

ST C3A gypsum calcium carbonate system **hydration**; delayed ettringite C3A gypsum calcium carbonate

IT **Hydration**, chemical
  Hydration kinetics
   (effects of gypsum content on delayed ettringite formation in **hydration** of 3CaO.Al2O3-CaSO4.2H2O-CaCO3 systems)

IT 12252-12-9, Ettringite (Ca6[Al(OH)6]2(SO4)3.xH2O)
   (delayed; effects of gypsum content on delayed ettringite formation in **hydration** of 3CaO.Al2O3-CaSO4.2H2O-CaCO3 systems)

IT 471-34-1, Calcium carbonate, processes 12042-78-3, Aluminum calcium oxide (Al2Ca3O6) 13397-24-5, Gypsum (Ca(SO4).2H2O), processes
   (systems; effects of gypsum content on delayed ettringite formation in **hydration** of 3CaO.Al2O3-CaSO4.2H2O-CaCO3 systems)

L70 ANSWER 8 OF 37 HCA COPYRIGHT 2003 ACS on STN

115:185630 Manufacture of improved wood materials with good flame retardance. Ochiai, Takeshi (Misawa Homes Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 03114801 A2 19910516 Heisei, 4 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 1989-254749 19890929.

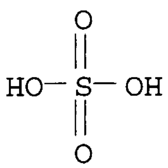
AB The title wood materials are manufd. by immersing wood in aq. solns. contg. water-sol. Ca salts, then in aq. solns. which generate water-insol. Ca compds. by chem. reaction of the Ca salts, then

immersing the wood in aq. solns. of metal salts at low concn. to **crystallize** the water-insol. Ca compds. at the surface of the wood and form mineral coating films. Thus, a Douflas fir board was immersed in water (100% water based on **bone** dry wt. was impregnated), immersed in aq. **CaCl2**, then in aq. (NH4)2SO4, dried, then immersed in 0.01 mol% Mg aq. soln., then dried to give a product showing noninflammability.

IT **7783-20-2**, Ammonium sulfate, uses and miscellaneous
10043-52-4, Calcium chloride, uses and miscellaneous
(aq. solns. of, wood immersed in)

RN 7783-20-2 HCA

CN Sulfuric acid diammonium salt (8CI, 9CI) (CA INDEX NAME)



2 NH_3

RN 10043-52-4 HCA

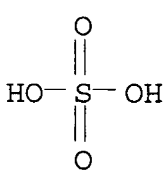
CN Calcium chloride (CaCl2) (9CI) (CA INDEX NAME)

Cl— Ca— Cl

IT **7778-18-9**, **Calcium sulfate**
(wood impregnated and coated with, for good fire resistance)

RN 7778-18-9 HCA

CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



● Ca

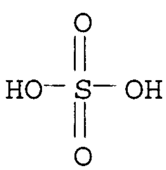
IC ICM B27K003-16
ICS B27K003-18

CC 43-2 (Cellulose, Lignin, Paper, and Other Wood Products)

IT 7439-95-4D, Magnesium, salts **7783-20-2**, Ammonium sulfate,

uses and miscellaneous **10043-52-4**, Calcium chloride, uses
and miscellaneous
(aq. solns. of, wood immersed in)
IT 471-34-1, Calcium carbonate, uses and miscellaneous 1305-62-0,
Calcium hydroxide, uses and miscellaneous **7778-18-9**,
Calcium sulfate
(wood impregnated and coated with, for good fire resistance)

L70 ANSWER 9 OF 37 HCA COPYRIGHT 2003 ACS on STN
115:73849 **Bone** and hide glues as fireproofing agents.
Hoelter, Heinz; Gresch, Heinz (Germany). Ger. Offen. DE 3941062 A1
19910620, 2 pp. (German). CODEN: GWXXBX. APPLICATION: DE
1989-3941062 19891213.
AB Solid, flammable materials, esp. wood and paper, are fireproofed by
treatment with 0.1-10% **bone** or hide glue, optionally
contg. **Na2SO4** or **CaCl2**.2H2O. The fireproofing
agents are harmless to the plant and animal world and are therefore
useful in extinguishing forest fires.
IT **7757-82-6**, Sodium sulfate, uses and miscellaneous
(**bone** and hide glue contg., as fireproofing agents)
RN 7757-82-6 HCA
CN Sulfuric acid disodium salt (8CI, 9CI) (CA INDEX NAME)



2 Na

IC ICM C09K021-00
ICS A62C039-00
CC 43-2 (Cellulose, Lignin, Paper, and Other Wood Products)
Section cross-reference(s): 45
IT Fireproofing agents
(**bone** and hide glues, for wood and paper)
IT Paper
Wood
(fireproofing agents for, **bone** and hide glue as)
IT Glues
(**bone**, fireproofing agents, for wood and paper)
IT **7757-82-6**, Sodium sulfate, uses and miscellaneous
10035-04-8, Calcium chloride **dihydrate**
(**bone** and hide glue contg., as fireproofing agents)

L70 ANSWER 10 OF 37 HCA COPYRIGHT 2003 ACS on STN
112:166043 The **hydration** kinetics of .alpha.-calcium sulfate

semihydrate. Bobrov, V. S.; Romashkov, A. V. (Ural. Nauchno-Issled. Proektn. Inst. Stroit. Mater., USSR). Izvestiya Akademii Nauk SSSR, Neorganicheskie Materialy, 26(1), 163-6 (Russian) 1990. CODEN: IVNMAW. ISSN: 0002-337X.

AB Different stages of the **hydration** of .alpha.-CaSO4.0.5H2O are described by using 3 independent equations. The H2O:gypsum ratio affects the no. of **crystals** of **CaSO4. 2H2O** formed during the **initial** stage of the reaction.

CC 67-3 (Catalysis, Reaction Kinetics, and Inorganic Reaction Mechanisms)
Section cross-reference(s): 58

ST **hydration** kinetics calcium sulfate hemihydrate

IT Kinetics of **hydration**
(of calcium sulfate hemihydrate)

IT 10034-76-1, Calcium sulfate (CaSO4) hemihydrate
(**hydration** kinetics of)

L70 ANSWER 11 OF 37 HCA COPYRIGHT 2003 ACS on STN

112:108801 Precipitation of calcium sulfate dihydrate at constant calcium activity. Klepetsanis, Pavlos G.; Koutsoukos, Petros G. (Res. Inst. Chem. Eng. Chem. Processes High Temp., Univ. Patras, Patras, GR-26110, Greece). Journal of Crystal Growth, 98(3), 480-6 (English) 1989. CODEN: JCRGAE. ISSN: 0022-0248.

AB At const. Ca activity at 25.degree., the kinetics of pptn. of CaSO4.2H2O was independent of pH, and an apparent order of reaction of n = 4, significantly different from the order of 2 found by seeded growth expts., suggested a polynucleation mechanism. The electrophoretic mobility of the CaSO4.2H2O particles showed a marked dependence on the soln. pH, which may affect the **crystal** growth of the **initially** formed **CaSO4. 2H2O crystallites** by altering the Ca2+/SO42- ratio on their surface.

CC 75-1 (Crystallography and Liquid Crystals)
Section cross-reference(s): 66, 68

ST calcium sulfate **hydrate** pptn; kinetics **crystn** calcium sulfate **hydrate**; electrophoretic mobility calcium sulfate **hydrate**

IT **Crystallization**
Crystallization kinetics
(of calcium sulfate dihydrate, at const. calcium activity)

L70 ANSWER 12 OF 37 HCA COPYRIGHT 2003 ACS on STN

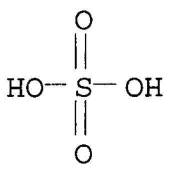
98:26832 **Dehydration** of selenite cleavage faces. Mehta, B. J. (Dep. Phys., Saurashtra Univ., Rajkot, 360/005, India). Crystal Research and Technology, 17(10), 1255-7 (English) 1982. CODEN: CRTEDF. ISSN: 0232-1300.

AB **Initial dehydration** of selenite (**CaSO4 .2H2O**) cleavage surfaces starts at 86.degree.. The dislocations are revealed by distd. H2O. Dislocations are mobile at **dehydration** temp. Impurity centers may act as obstacles for dislocation movement. **Dehydration** figures are probably

formed at impurity centers.
CC 78-9 (Inorganic Chemicals and Reactions)
ST selenite cleavage face **dehydration**; impurity center selenite **dehydration**; calcium sulfate dihydrate **dehydration**
IT **Dehydration**, chemical
(of calcium sulfate dihydrate cleavage faces)
IT 10101-41-4
(**dehydration** of cleavage faces of)

L70 ANSWER 13 OF 37 HCA COPYRIGHT 2003 ACS on STN
95:120027 Hemihydrate lime sulfate from phosphogypsum. Warachim, Henryk (Politechnika Gdanska, Pol.). Pol. PL 105200 19791115, 2 pp. (Polish). CODEN: POXXA7. APPLICATION: PL 1975-182141 19750716.
AB A mixt. of heated phosphogypsum and quicklime is mixed with concd. **H2SO4**. The **CaSO4**.cntdot.**2H2O** **initially** formed is **dehydrated** under a pressure of 0.2-0.6 mN/m2 at the liq./vapor equil. temp. of water to obtain the hemihydrate. The product is **dehydrated** by centrifuge and **dried** at >100.degree. or can be used directly in the manuf. of building materials. Suitable quantities are phosphogypsum 3 kg, quicklime 200 g, and concd. **H2SO4** 100 cm3.
IC C04B011-00
CC 58-3 (Cement and Concrete Products)

L70 ANSWER 14 OF 37 HCA COPYRIGHT 2003 ACS on STN
92:43984 Desulfurization of wet-process phosphoric acid. Okazaki, Takayoshi; Takakuwa, Yasuo; Nakajima, Sataro; Yozan, Tokumi (Nissan Chemical Industries, Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 54093694 19790724 Showa, 6 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 1977-158944 19771230.
AB Phosphate rock is decompd. with H3PO4 and **H2SO4**, the slurry contg. CaSO4.0.5H2O is cooled, treated with an aq. slurry of CaSO4.2H2O and CaHPO4, the mixt. kept at an appropriate temp. the **hydrate** the CaSO4.0.5H2O, and the ppt. is filtered and washed. Thus, Algerian ore (63% **bone** phosphate of lime) contg. P2O5 28.9 and **CaO** 48.62% 703 was stirred in a mixt. of impure H3PO4 from it and contg. P2O5 23.45 and **H2SO4** 2.70% 1725 and 75.1% **H2SO4** 785 g at 85-95.degree. for 30 min, the slurry 3052 g was cooled to 55.degree., treated with a mixt. of impure H3PO4 contg. P2O5 30.8 and **H2SO4** 1.98% 260.6 and the ore 36.8 g heated at 50.degree. for 1 h, the system filtered after 2 h, and the ppt. was washed with water. The product contained P2O5 31.27% (98.15% yield with 98.6% decompn.) and **H2SO4** 1.98%, vs. 30.50 (98.2 and 98.7%) and 3.50% when the slurry was kept at 55.degree. for 6 h, mixed with the slurry similarly prepd., and kept at 55.degree. for 2 h.
IT **7664-93-9**, uses and miscellaneous
(removal of, from wet-process phosphoric acid)
RN 7664-93-9 HCA
CN Sulfuric acid (8CI, 9CI) (CA INDEX NAME)



IC C01B025-22
CC 49-2 (Industrial Inorganic Chemicals)
IT 7664-38-2P, preparation
(purifn. of wet-process, **sulfuric acid** removal in)
IT **7664-93-9**, uses and miscellaneous
(removal of, from wet-process phosphoric acid)

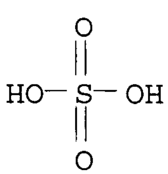
L70 ANSWER 15 OF 37 HCA COPYRIGHT 2003 ACS on STN
87:94776 Effect of the method of obtaining calcium sulfate hemihydrate on its vapor phase **hydration**. Triollier, Michel; Guilhot, Bernard (Cent. Chim. Phys., Ec. Super. Mines, Saint-Etienne, Fr.). Bulletin de la Societe Chimique de France (1-2, Pt. 1), 1-6 (French) 1977. CODEN: BSCFAS. ISSN: 0037-8968.
AB The effects of the methods of prepn. of CaSO4.0.5H2O on its subsequent vapor-phase **rehydration** to CaSO4.2H2O were studied by thermogravimetry, DTA, IR spectroscopy, and microcalorimetry. The **hydration** involves 3 steps: adsorption of H2O; dissoln. of the adsorbed H2O in the hemihydrate matrix; pptn. of a **new** phase, **CaSO4.2H2O**, when its soly. product in the solid phase is attained. It was possible to distinguish the processes of nucleation and **crystal** growth in the **rehydration**.
CC 78-9 (Inorganic Chemicals and Reactions)
ST calcium sulfate hemihydrate **hydration**
IT **Hydration**, chemical
(of calcium sulfate hemihydrate, effect of prepn. method of vapor-phase)
IT 10034-76-1
(**hydration** of, effect of prepn. method on vapor-phase)

L70 ANSWER 16 OF 37 HCA COPYRIGHT 2003 ACS on STN
83:137750 Hydration of calcium sulfate hemihydrate by water vapor. Triollier, Michel; Guilhot, Bernard (Cent. Chim. Phys., Ec. Natl. Super. Mines, St.-Etienne, Fr.). Comptes Rendus des Seances de l'Academie des Sciences, Serie C: Sciences Chimiques, 281(1), 27-9 (French) 1975. CODEN: CHDCAQ. ISSN: 0567-6541.
AB The rehydration of CaSO4.0.15H2O, obtained by vacuum **dehydration** of CaSO4.2H2O, by H2O vapor was followed by thermogravimetric anal. The mechanism comprises 3 steps: adsorption of H2O on the surface, dissoln. of the H2O in the matrix of the hemihydrate, and pptn. of a **new CaSO4.2H2O** phase once its soly. product in the solid phase is reached. The reaction is possible for pressures of H2O vapor less than the satn. pressure.

CC 68-2 (Phase Equilibriums, Chemical Equilibriums, and Solutions)
Section cross-reference(s): 78

L70 ANSWER 17 OF 37 HCA COPYRIGHT 2003 ACS on STN
77:79129 Effect of a gypsum-containing additive on the elimination of dust from furnace gases. Banit, F. G.; Vasilik, A. V. (USSR). Tsement (6), 17-18 (Russian) 1972. CODEN: TSMTAC. ISSN: 0041-4867.
AB The addn. of 3% of phosphoric acid by-product gypsum, contg. 95-8% **CaSO4.-2H2O**, to the **initial** high alkali charge in the manuf. of cement reduced the amt. of Cl-contg. compds. and increased the amt. of sulfates in the dust. This increased the effectiveness of the electrofilters. The content of dust in the gases was lowered. X-ray anal. showed that K in the dust was in the form of **K2SO4**.
CC 59-2 (Air Pollution and Industrial Hygiene)
Section cross-reference(s): 58

L70 ANSWER 18 OF 37 HCA COPYRIGHT 2003 ACS on STN
75:154592 Bleaching phosphate rock for use in ceramics. Smith, Raymond Anthony; Gilham-Dayton, Peter A. (Albright and Wilson (Mfg.) Ltd.). Brit. GB 1247861 19710929, 3 pp. (English). CODEN: BRXXAA. APPLICATION: GB 19680112.
AB A substitute for **bone** ash in ceramic compns. can be produced by bleaching of phosphate rock contg. small amts. of Cr and Fe in a process consisting of pelletizing, mixing in H3PO4 to which **H2SO4** was added in amts. of 5-15%, followed by a **calcination** at 1200-1350.degree.. Phosphate rocks most suitable for bleaching contain Fe as Fe2O3 <0.2 wt. % and Cr as Cr2O3 <45 ppm. During, before, or after the pelletization sufficient H3PO4 is added to adjust the **CaO**:P2O5 molar ratio to (3.0-3.3):1; 50-150 g **H2SO4** is added per 1. of H3PO4 and the mixt. is **calcined** at 1200-1350.degree. in the presence of water vapor.
IT **7664-93-9**, uses and miscellaneous
(in bleaching, of phosphate rock)
RN 7664-93-9 HCA
CN Sulfuric acid (8CI, 9CI) (CA INDEX NAME)



IC C01B
CC 57 (Ceramics)
ST bleaching phosphate rock; **bone** ash substitute ceramics; phosphoric acid bleach phosphate rock; iron removal phosphate rock; chromium removal phosphate rock
IT Ceramic materials
(bleaching of phosphate rock for, phosphoric acid and

sulfuric acid in)

IT Phosphate rock
(bleaching of, phosphoric acid and **sulfuric acid** in)

IT Bleaching
(of phosphate rock, phosphoric acid and **sulfuric acid** in)

IT 7664-38-2, uses and miscellaneous **7664-93-9**, uses and miscellaneous
(in bleaching, of phosphate rock)

L70 ANSWER 19 OF 37 HCA COPYRIGHT 2003 ACS on STN

69:6819 Luminescence and exoemission of sulfate phosphors after ionizing irradiation. Krasnaya, A. R.; Yaskolko, V. Ya. Izvestiya Akademii Nauk SSSR, Seriya Fizicheskaya, 32(1), 44-6 (Russian) 1968. CODEN: IANFAY. ISSN: 0367-6765.

AB The thermostimulated luminescence (TL) and exoemission (TE) of CaSO4 after irradn. with .beta.-particles from 90Sr were detd. by the previous method (loc. cit.). CaSO4 was prepd. from **CaCl2** and **H2SO4** with different stoichiometric deficiencies. Some were activated with Mn++ and Sm+++. In crystals with an insufficiency of **H2SO4**, activated and nonactivated, 3 peaks with Tmax. .apprx. 125, 200, and 275.degree. of TE appeared. Activation with Mn++ lowered the TL peaks, whereas activation with Sm extinguished the old peaks and developed new peaks. The relative growth of the TE peaks in crystals with insufficient **H2SO4** could not be accounted for by the pH of the medium nor by the effect of Cl-. The Mn in the crystals is in the Mn4+ form. There is a difference between the TL and TE capture centers. The properties of these centers depend on the prehistory of the **initial CaSO4.2H2O**.

CC 73 (Spectra and Other Optical Properties)

L70 ANSWER 20 OF 37 HCA COPYRIGHT 2003 ACS on STN

69:4027 CGC process-method for manufacturing wet-process phosphoric acid. Hayakawa, Masashi (Res. Develop. Dep., Central Glass Co., Ltd., Ube, Japan). Japan Chemical Quarterly, 4(2), 26-9 (English) 1968. CODEN: JCHQAS. ISSN: 0448-8571.

AB The CGC process increases H3PO4 yield while completely eliminating the H3PO4 content in the by-product CaSO4, thereby increasing its value. The process, a dihydrate-hemihydrate process, consists of 2 steps, H3PO4 manuf. and gypsum reforming. In the H3PO4 step of the **new** process, H3PO4 and **CaSO4.2H2O** are produced in the attacking tank by decompg. pulverized phosphate rock with a mixt. of **H2SO4** and H3PO4 which is circulated from the gypsum reforming step. Ca3(PO4)2 + 3H2SO4 + 6H2O .fwdarw. 2H3PO4 + 3CaSO4.2H2O. The slurry is sepd. without any cake washing into H3PO4 and CaSO4.2H2O. The cake contg. H3PO4 is sent to the gypsum reforming step, while the filtrate, as product H3PO4, is used for fertilizer manuf. and other industrial purposes. In the reforming section, the cake is mixed in the reforming tank with **H2SO4** to form a slurry, the temp. of which is raised

sufficiently to convert the dihydrate to hemihydrate: CaSO4.2H2O .fwdarw. CaSO4.0.5H2O + 1.5H2O. Conversion temp. ranges from 70.degree. to 85.degree., depending on the concn. of the **H2SO4**, the type of phosphate rock, etc. As the dihydrate is recrystd. into hemihydrate, the H3PO4 in the dihydrate exudes into the liq. phase and any undecompd. phosphate rock is decompd. by the **H2SO4**. The hemihydrate slurry is filtered and the filtrate is returned to the attacking tank. Since the reformed CaSO4.0.5H2O is obtained as large crystals, it lends itself to easy filtration and washing. The crystals are so stable that washing with water at room temp. does not cause the hemihydrate to be converted to dihydrate. Gypsum anal. data and results of cement tests are presented.

CC 49 (Industrial Inorganic Chemicals)

IT 7664-38-2P, preparation
(wet-process, from phosphate rock with phosphoric acid and **sulfuric acid**)

L70 ANSWER 21 OF 37 HCA COPYRIGHT 2003 ACS on STN

68:24342 Effect of electrylyte additives on **hydration** hardening of mineral binders. Segalova, E. E.; Kontorovich, S. I.; Shabanova, E. A. Fiz.-Khim. Mekh. Pochv, Gruntov, Glin Stroit. Mater. 319-24 From: Ref. Zh., Khim. 1967, Pt. II, Abstr. No. 17M166 (Russian) 1966.

AB The effect of electrolytes not reacting with CaSO4.0.5H2O and **CaO** on **hydration** hardening of these binders was assocd. with an increase in the size of the **crystals** of the **new** phase (**CaSO4.2H2O** and Ca(OH)2) and with a decrease in internal tensile forces. In suspensions of CaSO4.0.5H2O, addns. of electrolytes decreased the final strength, whereas addn. of electrolytes to a **CaO** suspension generally increased strength.

CC 58 (Cement and Concrete Products)

ST ENING; ELECTROLYTES MINERAL BINDERS HARD; MINERAL BINDERS **HYDRATION** HARDEN; **HYDRATION** HARDENING MINERAL BIND

IT Electrolytes, properties
(**hydration** of lime and plaster of Paris contg.)

IT Lime
(**hydration** of, electrolyte effect on)

IT **Hydration**, chemical
(of plaster of Paris, electrolyte effect on)

IT 26499-65-0
(**hydration** of, electrolyte effect on)

L70 ANSWER 22 OF 37 HCA COPYRIGHT 2003 ACS on STN

58:37880 Original Reference No. 58:6439h,6440a Physico-chemical investigation of some **crystal hydrates**. Berg, L. G.; Saibova, M. T. Uzbekskii Khimicheskii Zhurnal, 6(No. 5), 54-60 (Unavailable) 1962. CODEN: UZKZAC.

AB Thermographic studies are presented on the thermal decompn. of gypsum (CaSO4H2O), astrakanite (**Na2SO4**.MgSO4.4H2O), and carnallite (KCl.MgCl26H2O) at 25-760 mm. These studies show

establishment of a dissocn. pressure dependence on temp. for the given **cryst. hydrates**. Heats of dissocn. and some other thermodynamic consts. for the dissocn. reactions are calcd. The temp. effect of the reaction, which is accompanied by the sepn. of a gas phase, does not depend on pressure, e.g., CaSO4.2H2O = CaSO4.0.5H2O + 1.5H2O (200-760 mm.), **Na2SO4**.MgSO4.4H2O = **Na2SO4**.MgSO4.2H2O + 2H2 (25-760 mm.). This deviation can be explained by dual processes accompanying each corresponding reaction (1) decompn. of the **initial** phase (**CaSO4.2H2O**, **Na2SO4**.MgSO4.4H2O); (2) removal of the H2O formed in the decompn. process as a gas. Hence, the breakdown of the initial salt into two phases predet. the character of the whole process. Consequently, in heating **cryst. hydrates** of salts, it cannot be assumed that if the temp. does not change with change in pressure that it is always an effect due to the melting process.

CC 14 (Inorganic Chemicals and Reactions)

IT **Dehydration**
Heat of **dehydration**
(of bloedite, carnallite and gypsum)

IT Entropy
(of **dehydration** of bloedite, carnallite and gypsum)

L70 ANSWER 23 OF 37 HCA

56:58866 Original Reference No. 56:11195i,11196a Kinetics of endothermic reactions in minerals. I. **Dehydration** of gypsum. Portoles, J. L. Amoros; Galvan, J.; Alonso, P. (Lucas Mallado Inst., Madrid). Bol. Real. Soc. Espan. Hist. Nat., Secc. Geol, 59(1), 13-23 (Unavailable) 1961.

AB When heated, disorientation of gypsum fibers begins at 85.degree.. Sol. anhydrite, coexisting with gypsum, begins to form at 105.degree., and the formation is complete at 110.degree.. This form is stable to .apprx.275.degree., where the anhydrite begins to form. The kinetics of the reaction are discussed in terms of the crystal structure of the **initial** substance, **CaSO4.2H2O**, and the final product, CaSO4.

CC 14 (Inorganic Chemicals and Reactions)

IT Reaction kinetics and(or) Velocity
(of **dehydration**, of gypsum)

IT **Dehydration**
(of gypsum)

IT 13397-24-5, Gypsum
(**dehydration** of)

IT 14798-04-0, Anhydrite
(formation of, in gypsum **dehydration**)

L70 ANSWER 24 OF 37 HCA

53:82508 Original Reference No. 53:14850i Preliminary identification of crystalline phases in a transparent stalactite. Benington, Fred (Battelle Mem. Inst., Columbus, O.). Science (Washington, DC, United States), 129, 1227 (Unavailable) 1959. CODEN: SCIEAS. ISSN: 0036-8075.

AB Of two cryst. phases found in a cavern stalactite from newly explored passages in the Flint Ridge Cavern system of south-central Kentucky, the major phase is mirabilite, whereas the minor phase, according to preliminary data (Na:Ca = 3.82, n 1.518), is a **new** mineral, 2 **Na2SO4.CaSO4.2H2O**
, which is stable at temps. above 25.degree..

CC 8 (Mineralogical and Geological Chemistry)

L70 ANSWER 25 OF 37 HCA COPYRIGHT 2003 ACS on STN

52:65656 Original Reference No. 52:11750g-i,11751a-i,11752a-c The methylation of N-acetylglucosamine derivatives. Kuhn, Richard; Baer, Hans Helmut; Seeliger, Annemarie Ann., 611, 236-41 (Unavailable) 1958.

AB With MeI and BaO in HCONMe2 permethylation in 1 operation is possible; good yields of the O-Me ether of .beta.-Et and .alpha.-benzyl-N-acetyl-D-glucosaminide as well as of N-acetyllactosaminol are obtained. Thus, in a 3-necked flask with stirrer was refluxed 5 g. .beta.-ethyl-N-acetyl-D-glucosaminide in 50 cc. HCONMe2 (**dried** over BaO), 15 cc. MeI, and 18.4 g. finely powd. BaO, the spontaneous rise in temp. being controlled by stirring and cooling so that the temp. was kept at 40-5.degree.. After another hr. the temp. fell to 23.degree. and stirring was continued 5.5 hrs. The resulting thin, yellow paste added to 500 cc. CHCl3, filtered by suction, and shaken 3 times with 100 cc. H2O became colorless; the washings extd. 3 times with 50-cc. portions of CHCl3, **dried** over Na2SO4, and evapd. in vacuo yielded 4.8 g. **cryst**. .beta.-Et 3,4,6-trimethyl-N-acetyl-D-glucosaminide (I), m. 190-1.degree. (EtOAc), [.alpha.]20D 5.9.degree. and 5.6.degree. (different prepns.) (c 2, CHCl3), -16.7.degree. (c 2, MeOH), unchanged by **recrystn**. from C6H6. A similar expt. carried out without cooling resulted in a temp. rise in 1.5 hrs. to 87.degree., which dropped slowly, and after 3.5 hrs. was obtained 4.7 g. crude product yielding on **recrystn**. from EtOAc 3.3 g. product, m. 191.degree.. When HCONMe2 (contg. 0.5% H2O) was used, the crude yield was 5.6 g.; the temp. rose to 95.degree. after 52 min., fell to 66.degree., and the mixt. was shaken 3.5 hrs. The **recrystd**. product (3.2 g.) m. 191-2.degree.. I (500 mg.) refluxed 15 hrs. with 50 cc. N HCl, treated with **bone** black in vacuo at a low temp., and washed many times with H2O before **drying** gave 280 mg. 3,4,6-tri-O-methyl- D-glucosamine-HCl (II), becoming brown at 200.degree. without melting (MeOH-Et2O), [.alpha.]20D 51.9.degree. (initial) .fwdarw. 99.6.degree. (c 1, H2O). N-Acetyllactosamine (6 g. synthetic product, mol. wt. 415 with 1 MeOH) in 50 cc. H2O treated with 1.1 g. KBH4, kept at room temp. 2 hrs. until a slightly acidified test soln. no longer reduced Fehling soln., the K ions removed by Amberlite IR-120(H+), the excess KBH4 decompd. (very little H evolution), and the mixt. evapd. to **dryness**, first in vacuo, then treated many times with MeOH until no green color (test for B) was obtained yielded 5.4 g. N-acetyllactosaminol (III), C14H27NO11. III (1.50 g.) in 20 cc. **dried** HCONMe2 was treated with 13.3 g. MeI (3 times theory) and 7.2 g. finely

powd. BaO and shaken under anhyd. conditions; the mixt. became warm gradually at first, then the temp. rose suddenly after about 1 hr. and cooling was necessary to maintain it at 40-5.degree.; after 50-60 min., the action slackened and the reaction was completed in 4-5 hrs. The process described for the prepn. of II was carried out giving 1.75 g. crude octamethyl-N-acetyllactosaminol (1,3,5,6-tetra-O-methyl-2-deoxy-2-acetamido-4[2,3,4,6-tetra-O-methyl-D-galactopyranosyl]-D-sorbitol (IV), b. 200-10.degree. (bath temp.); the pure product (1.50 g. after many distns.) b0.001 205-10.degree., [.alpha.]20D -18.6.degree.(c 1.4, CHCl3), n20D 1.4672. Acetyl-D-glucosamine (30 g.) in 120 cc. PhCH2OH (distd. over **CaO**, contg. 0.5% HCl) heated to boiling under reflux 30 min., **dry** Et2O added to the cooled soln. with vigorous shaking, the dark oily ppt. removed from the first Et2O ext. by decantation, and 2.5-4 vols. Et2O added pptd. 30 g. light brown powder, which filtered off, washed several times with Et2O, and **recrystd**. from about 150 cc. hot EtOH yielded about 18 g. .alpha.-benzyl-N-acetyl-D-glucosaminide (V), m. 183-4.degree. (EtOH), [.alpha.]23D 168.5.degree. (c 1, H2O) [the corresponding .beta.-form m. 205-6.degree., [.alpha.]20D -48.degree. (H2O)(C.A. 49, 2332f)]. .alpha.-Benzyl glucoside (10 g.) and 10 g. finely powd. anhyd. ZnCl2 dissolved under anhyd. conditions in 35 cc. BzH at 60.degree., shaken 7-10 min., then kept at the same temp. 30 min., shaken well with 3 vols. H2O, and the ppt. from the brown reaction mixt. filtered by suction, washed with H2O, then 20 cc. EtOH, and digested with 200 cc. **dry** Et2O yielded 9.4 g. almost colorless crude product, which **recrystd**. from 120 cc. hot C5H5N followed by 1-1.5 vols. hot H2O, and cooled yielded fine needles which were washed with 60 cc. C5H5N-H2O then with H2O. The yield of .alpha.-benzyl N-acetyl-4,6-benzylidene-D-glucosaminide (VI) was 6.7-7.9 g., after 2 more **crystns**. from C5H5N-H2O, VI m. 262.degree. (decompn.), [.alpha.]23D 114.degree. (c 1.1, C5H5N). VI (150 mg.) dissolved in 2 cc. **dry** HCONMe2 with warming, treated with 0.5 cc. MeI and 0.5 g. BaO, shaken occasionally at room temp. 30 min., then warmed on a water bath 2 hrs. under reflux at 40-5.degree., the paste stirred occasionally with a glass rod, the excess MeI removed in vacuo after 2 hrs. on the gradually cooling water bath, the residue treated with 10 cc. ice H2O contg. a drop of phenolphthalein soln., stirred well while dil. HCl was added until all particles were free from alkali, and the yellow **cryst**. residue quickly filtered off, washed with H2O, and covered with C5H5N to remove the brown color formed by air gave 149 mg. **cryst**. .alpha.-benzyl-N-acetyl-3-(O-methyl)-3,4,6-benzylidene-D-glucosaminide (VII), **recrystd**. from hot C5H5N and a little hot H2O and **dried** (130.degree./3 mm., P2O5) yielding 133.5 mg. product, colorless needles, m. 272.degree., [.alpha.]20D 96.degree. (c 1, C5H5N); after repeated **recrystn**. it m. 273.degree. (PhMe), 271.degree. (BuOH). VI (5.8 g.) in 50 cc. abs. C5H5N cooled to -20.degree., treated dropwise with 3.5 cc. BzCl, kept 20 min. at -20.degree. and 15 hrs. at 4.degree., then dild. with 500 cc. CHCl3, shaken 3 times with ice water, ice-cold 2N **H2SO4**, satd. NaHCO3 soln., and

again with ice water, the org. phase **dried** with Na2SO4 and the white, **cryst**. solid, **recrystd**. from 250 cc. C6H6 yielded 5.95 g. .alpha.-benzyl-N-acetyl-3-benzoyl-4,6-benzylidene-D-glucosaminide (VIII), m. 218-20.degree., [.alpha.]21D 44.degree. (c 1, C5H5N); by the addn. of ligroine, 0.6 g. was recovered from the mother liquor, giving a total of 90%. VIII (5.9 g.) dissolved in 180 cc. AcOH on the steam bath, treated under reflux with 120 cc. H2O, heated 30 min., the BzH split off, and AcOH removed in vacuo (bath temp. 40.degree.), and the residue then evapd. many times with H2O and PhMe yielded from C6H6 3 g. .alpha.-benzyl-N-acetyl-3-benzoyl-D-glucosaminide (IX), [.alpha.]23D 104.degree. (C5H5N). The first crude product, [.alpha.] 96.degree., m. 80-3.degree., after several **recrystns**. from C6H6, m. 95-7.degree., [.alpha.]23D 106.degree. (c 1, C5H5N). IX (750 mg.) was shaken with 20 cc. HCONMe2, 11 cc. MeI, and 10 g. Ag2O 40 hrs. at room temp., centrifuged, the solid phase washed twice with HCONMe2, the soln. treated with 5 vols. CHCl3, kept overnight at 4.degree., the ppt. of Me4NI.2AgI filtered off, and the filtrate shaken 4 times with H2O, **dried**, evapd. in vacuo, and the oily residue chromatographed and eluted with ligroine (b. 70-80), 1:1 ligroine-Et2O, and 1:1 Et2O-Et2Ac; the Et2O contained 176 mg. optically active product, [.alpha.]25D 105.degree. (c 2, CHCl3). After removal of the Bz group with MeOHNH3, a sublimate of BzNH3 was obtained at 100.degree./10-3 mm. and also a distillate at 180.degree., giving an unsatisfactory analysis for benzyldi(O-methyl)-N-acetylglucosaminide (X). In another expt. 3 g. IX was shaken with 8 cc. HCONMe2, 30 cc. MeI, and 30 g. Ag2O 15 hrs. at room temp., and the pasty mixt. treated with excess CHCl3 and worked up as before. Half of the product from CHCl3 was distd. in a high vacuum immediately and an appreciable amt. of HCONMe2 appeared in the receiver; 2 distns. (5 .times. 10-3 mm., 160-180.degree. bath temp.) gave 0.95 g. .alpha.-benzyl-N-acetyltrimethylglucosaminide, colorless oil, specific rotation 153.degree. (c 2, CHCl3), C18H27NO6, which soon **crystd**.; **recrystn**. from 4:1 Et2OMeOH gave a product, m. 151-2.degree. (sintering at 147.degree.), [.alpha.]23D 148.degree. (c 2 and 0.5, CHCl3).

CC 10 (Organic Chemistry)

L70 ANSWER 26 OF 37 HCA COPYRIGHT 2003 ACS on STN

41:36811 Original Reference No. 41:7292h-i,7293a-c Chemistry of calcium orthophosphates. Herbert, Claude (Ecole nationale superieure Mines, Paris). Ann. mines & carburants, Mem. 136(No. 4), 5-88 (Unavailable) 1947.

AB Mineral phosphates from all parts of the world and beef-**bone** phosphates have been examd. spectroscopically for trace elements. Most of them contain Ag, Pb, Cu, Cd, As, Sn, Sb, Mo, Fe, Cr, Al, Ti, Mn, Zn, Zr, Be, Mg, Sr, Si, Na, K, Li. Some also contain Au, Pt, Pd, V, W, Ga, Ta, Ba, Rb. All showed some radioactivity (equiv. to 0.03 to 0.69 mg. U per g. phosphate). Cd, Zr, and Sr were present in the minerals but lacking in the **bone** phosphates. Solvent action of acids on these phosphates, as revealed by electrometric pH titration and by elec. cond., can differentiate

between high- and low-soly. phosphates if the acid concn. and the acid/phosphate ratio are properly chosen. A moderately weak acid (H_3PO_4, citric, acetic) gives greater differences than either strong (HCl, HNO_3, **H_2SO_4**) or very weak acids, such as boric acid, which does not attack pptd. $Ca_3(PO_4)_2$. Of those tested, AcOH gave the greatest differences. H_2SO_3 also attacks Ca phosphates, but the reaction is complicated by atm. oxidation of sulfite to sulfate. A simple method by prepg. $CaHPO_4.2H_2O$ involves neutralizing one l. of milk of lime contg. 4.4 g. **CaO** with 85% H_3PO_4 in 22% excess. The mixt. is stirred for two days, during which time the pH rises from 4 to 4.6-4.8. The **crystals** are centrifuged off, washed with EtOH, and air-**dried**. Use of a smaller excess of H_3PO_4 gives too-basic a product. The **dihydrate** is readily converted to $CaHPO_4$ by boiling a suspension for 30 min., giving a product contg. 7.8% H_2O instead of the theoretical 6.6% H_2O.

CC 6 (Inorganic Chemistry)

L70 ANSWER 27 OF 37 HCA

41:13834 Original Reference No. 41:2821b-i,2822b-f American Society for Testing Materials, Standards, 1946. II. Nonmetallic materials, constructional. 1762 pp. (Unavailable) 1946.

AB cf. C.A. 39, 2588.4; 40, 4821.9. Standards adopted or revised in 1946 are given for: portland cement and its chem. analysis; sampling hydraulic cement; test for compressive strength of natural building stone; test for drying and firing shrinkage of fireclay plastic refractories; test for crushing strength and modulus of rupture of insulating fire brick at room temp.; test for permanent linear change on reheating of insulating firebrick; test for pyrometric cone equiv. of refractory materials; test for permanent linear change after reheating of refractories; tests for apparent porosity, water absorption, apparent sp. gr., and bulk d. of burned refractory brick; tests for sieve analysis and water content of refractory materials; terms relating to refractories; symbols for heat transmission; concrete aggregates; test for abrasion of coarse aggregate by use of the Los Angeles machine and the Deval machine; measuring of mortar-making properties of fine aggregate; test for sieve analysis of fine and coarse aggregates; slow-setting emulsified asphalt; cut-back asphalt; test for water in petroleum products and other bituminous materials; definitions of terms relating to materials for roads and pavements; test for flash and fire points by means of open cup; wide-selvage asphalt roofing surfaced with mineral granules; asphalt shingles and asphalt siding surfaced with mineral granules; lampblack; **bone** black; carbon black; iron blue; ultramarine blue; pure chrome green; chrome oxide green; permanently liquid oiticica oil; isopropyl alc.; Me Et ketone; tritolyl phosphate; test for distn. of gasoline, naphtha; kerosene, and similar petroleum products; sampling and testing of lacquer solvents and diluents; test for light sensitivity of traffic paint, conducting of road service tests on traffic paint; definitions of terms relating to paint, varnish, lacquer, and related products; test for combustible properties of treated wood by

the crib test method; A.S.T.M. thermometers. Tentative standards issued or revised in 1946 are given for: air-entraining portland cement; portland blast-furnace slag cement; test for air content of portland cement mortar; chem. analysis of portland cement; test for fineness of portland cement by air-permeability app.; normal-finishing and special-finishing **hydrated** lime; **hydrated** lime for masonry purposes; structural clay facing tile; definitions of terms relating to structural clay tile; structural insulating board made from vegetable fibers and its testing; test for soundness of aggregates by use of **Na2SO4** or MgSO4; definitions of terms relating to concrete and concrete aggregates; **CaCl2** and its sampling and testing; sampling stone, slag, gravel, sand, and stone block for use as highway materials; sampling bituminous materials; test for sulfonation index of road tars; asphalt roofing surfaced with mineral granules and with powd. talc or mica; TiO2 pigments; pumice pigment; red and brown iron oxide pigments; **hydrated** yellow iron oxide; chem. analysis of Zn yellow pigment (Zn chromate yellow); testing of cellulose acetate; test for ester value of tritolyl phosphate; evaluating degree of resistance of traffic paint to bleeding; test for dry to no-pick-up time of traffic paint; evaluating degree of setting of traffic paint; prepn. of steel panels for testing paint, varnish, lacquer, and related products; test for changes in protective properties of coatings of paint, varnish, lacquer, and related products on steel surfaces when subjected to immersion; operating light- and water-exposure app. (carbon-arc type) for testing paint, varnish, lacquer, and related products; test for volatile oil in rosin; sampling and testing of dipentene; test for water in liquid naval stores; sampling and testing pine oil; sampling and testing pine tars and pine-tar oils; terms relating to naval stores and related products; test for combustible properties of treated wood by the fire-tube method; A.S.T.M. thermometers; detn. of pH of aq. solns. with the glass electrode. Tentative revisions, submitted in 1946, of standards are given for: chem. analysis of portland cement; test for compressive strength of hydraulic-cement mortars; building brick made from clay or shale; sand-lime building brick; sampling and testing brick; structural clay tile and its sampling and testing; refractories for stationary boiler service; fireclay plastic refractories for boiler and incinerator services; refractories for incinerators; refractories for malleable Fe furnaces with removable bungs and for annealing ovens; panel test for resistance to thermal and structural spalling of refractory brick, of high-heat-duty fireclay brick, and of fireclay plastic refractories; test for workability index of fireclay plastic refractories; test for true sp. gr. of burned refractory materials; definitions of terms relating to refractories; TiO2 pigments; pure Zn yellow (Zn chromate); specification and tests for sol. nitrocellulose; sampling and testing lacquer solvents and diluents; fire tests of building construction and materials; A.S.T.M. thermometers.

IT **10043-52-4**, Calcium chloride
(sampling and testing of, standards for)

RN 10043-52-4 HCA
CN Calcium chloride (CaCl2) (9CI) (CA INDEX NAME)

Cl—Ca—Cl

CC 13 (Chemical Industry and Miscellaneous Industrial Products)
IT Charcoal
(**bone** black or animal, standards for)
IT Lime
(**hydrated** or slaked, standards for)
IT **10043-52-4**, Calcium chloride
(sampling and testing of, standards for)

L70 ANSWER 28 OF 37 HCA COPYRIGHT 2003 ACS on STN
40:14010 Original Reference No. 40:2666a-b Hydrolysis of starch to dextrose. Fetzer, Walter R. (Union Starch & Refining Co.). US 2393095 19460115 (Unavailable). APPLICATION: US .
AB In a cyclic process for the production of dextrose the accumulation of ash in the liquor is avoided by hydrolyzing the starch with **H2SO4** and neutralizing with **CaO**, followed By filtration through **bone** char to remove **CaSO4**. After repeated evapn. and **crystn**. of the dextrose, the residual **CaSO4**-free soln. of reversion products is mixed with a fresh starch suspension for a new hydrolysis cycle. Cf. C.A. 35, 342.6.
CC 28 (Sugar, Starch, and Gums)

L70 ANSWER 29 OF 37 HCA COPYRIGHT 2003 ACS on STN
36:19152 Original Reference No. 36:2952g-i,2953a-i,2954a American Society for Testing Materials, Standards, 1941 Supplement. II. Non-metallic materials, constructional. 426 pp. (Unavailable).
AB Standards adopted or revised in 1941 are given for portland cement; gypsum; gypsum partition tile or block; definition of terms relating to gypsum; structural clay load-bearing wall tile and non-load-bearing tile; sampling and testing brick; fire tests of building construction and materials and of door assemblies; refractories for heavy- and for moderate-duty stationary boiler service, for incinerators, for malleable-iron furnaces with removable bungs and for annealing ovens; ground fire clay; classifications of fire-clay refractories; load test at high temps. of fire-clay refractories; testing insulating firebrick for compressive strength, flexural strength and permanent linear change after heating; tests for size and bulk d. of refractory brick; test for warpage of refractory brick and tile; tests for apparent porosity, water absorption, apparent sp. gr. and bulk d. of burned refractory products; chem. analysis of refractory materials; definitions of symbols for heat transmission; concrete sewer pipe; reinforced concrete sewer pipe and culvert pipe; preformed expansion joint fillers for concrete and methods for testing them; test for sieve analysis of mineral filler; asphalt for use in constructing built-up roof coverings; coal-tar pitch for roofing, dampproofing

and waterproofing and creosote for priming coat with the pitch; primer for use with asphalt in dampproofing and waterproofing; asphalt mastic for use in waterproofing; bituminous grouts for use in waterproofing below and above ground level; coal-tar-satd. roofing felt for use in waterproofing and in constructing built-up roofs; testing of bituminous mastics, grouts and like mixts.; tests for coarse particles in mixts. of asphalt and mineral matter; test for tar acids in creosote and creosote-coal tar solns.; definitions of terms relating to timber preservatives; lithopone (ZnS pigments); TiO2 pigments; basic carbonate white lead; basic sulfate white lead; Zn oxide; leaded Zn oxide; blue lead, basic sulfate; **bone** black; C black; lampblack; mineral Fe oxide; ocher; Prussian blue; ultramarine blue; pure and reduced chrome greens; chrome oxide green; chrome yellow; c. p. zinc yellow (zinc chromate); c. p. para red toner; reduced para red; red lead; Al powder and pigment paste for paints; gold bronze powder; zinc dust; raw or refined perilla oil; raw soybean oil; raw tung oil; testing of drying oils; orange shellac; shellac varnishes; sampling and testing lacquer solvents and diluents; test for elongation of attached lacquer coatings with the conical mandrel test app.; test for consistency of enamel-type paints; and chem. analysis of white linseed oil paints. Tentative standards issued or revised in 1941 are given for turbidimetric test for fineness of portland cement; definitions of terms relating to lime and to gypsum; building brick made from clay or shale; vitrified-clay filter block for trickling filters; test for compressive strength of natural building stone; mortar for reinforced brick masonry; test for fire-retardant properties of wood; sampling and prepn. of specimens for testing of thermal insulating cement; tests for vol. change upon drying, covering capacity and bulk d. of thermal-insulating cement; tests for compressive and flexural strengths of preformed block-type, and thickness and d. of blanket-type, thermal-insulating materials; definition of terms relating to thermal-insulating materials; classification of insulating back-up block and insulating firebrick; chem. analysis of soda-lime glass; definition of the term glass; ready-mixed concrete; test for soundness of aggregates by use of **Na2SO4** or MgSO4; coarse aggregate for highway construction; crushed slag and stone for bituminous macadam base and surface courses; NaCl for road purposes; slow-setting emulsified asphalt for fine aggregate mixes; sampling bituminous materials; tests for modified miscibility and cement mixing of emulsified asphalts; vol.-correction table for tar and coal-tar pitch; asphalt-satd. roofing felt for use in waterproofing and in constructing built-up roofs; asphalt roofing and shingles surfaced with mineral granules or powd. talc or mica; Al silicate pigment; BaSO4 pigment; diatomaceous silica pigment; lead titanate; Mg silicate pigment; mica pigment; sampling and testing of Al powder and paste; oiticica oil; liquid paint driers; test for color of orange shellac; testing of liquid driers; dibutyl phthalate; prepn. of steel panels for exposure tests of enamels; evaluation of degree of resistance to rusting obtained with paint on Fe or steel; test for specular gloss of paint finishes; and definitions of terms relating to paint,

varnish, lacquer and related products. Tentative revisions of standards are submitted for quicklime and **hydrated** lime for silica brick manuf.; testing gypsum and gypsum products; panel tests for resistance to thermal and structural spalling of refractory brick, high-heat-duty fire-clay brick and super-duty fire-clay brick; test for pyrometric cone equiv. of refractory materials; clay sewer pipe; concrete aggregates; test for abrasion of coarse aggregate by use of the Los Angeles machine; tests for sp. gr. and absorption of coarse and of fine aggregates; test for structural strength of fine aggregate using const. water-cement-ratio mortar; securing specimens of hardened concrete from the structure; making and storing compression test specimens of concrete in the field; tests for compressive strength of concrete; chem. analysis of **CaCl2**; test for ductility of bituminous materials; asphalt-satd. asbestos felt for use in constructing built-up roofs; and sampling and testing turpentine.

IT **10043-52-4**, Calcium chloride
(specifications or standards for)

RN 10043-52-4 HCA

CN Calcium chloride (CaCl2) (9CI) (CA INDEX NAME)

Cl—Ca—Cl

CC 13 (Chemical Industry and Miscellaneous Industrial Products)

IT Charcoal
(**bone** black or animal, specifications or standards for)

IT 7439-89-6, Iron **10043-52-4**, Calcium chloride 13530-65-9, Zinc chromate
(specifications or standards for)

L70 ANSWER 30 OF 37 HCA COPYRIGHT 2003 ACS on STN

14:409 Original Reference No. 14:95h-i,96a-d Treatment of boronatrocalcite. Longobardi, E.; Camus, N. Anales soc. quim. Argetina, 7, 12-21 (Unavailable) 1919.

AB The Eckelt method of treating boronatrocalcite for recovery of H3BO3 was tested and found not suitable for the treatment of Argentine deposits of this mineral, owing largely to the relatively low concn. and excessive cost of the HCl employed. **H2SO4** was substituted for HCl and a method involving the following steps was worked out: (1) grinding of the mineral, (2) liberation of H3BO3, (3) hot filtration, (4) preliminary **crystn.**, (5) washing, (6) **recrystn.** The pulverized mineral and 90% **H2SO4** were gradually added, either alternately or simultaneously in small portions, to a certain amt. of H2O (4-5 times the wt. of the mineral under treatment) contained in a receptacle lined with Pb and equipped with a stirring app.; the temp. was maintained at 80-90.degree. during the operation. Excess of acid must be avoided and the amt. of free **H2SO4** at the end of the operation should not exceed 0.10-0.25%. The boronatrocalcite employed contained an av. of 7.25% Na2O, 11.5% **CaO** and 34% B2O3 with admixt. of various salts, especially

CaSO4, Na2SO4 and NaCl. Slightly more than 0.5 kg. of 90% **H2SO4** was required for each kg. of **hydrated** B2O3 obtained. The mass obtained after treatment with **H2SO4** was filtered (at 70-80.degree.) by means of a filter press and the filtrate was then cooled; the **crystd**. H3BO3 thus obtained contained small ants. of NaCl, Na2SO4, etc. The latter were almost entirely eliminated by washing the H3BO3 with cold H2O and **recrystg**. In order to obtain **crysts**. of prismatic form the washed H3BO3 was **recrystd**. by dissolving it in H2O at 80-90.degree. so as to obtain a soln. of 1.040-1.050 sp. gr. (at 90.degree.); a small amt. (0.05-0.1%) of **H2SO4** was then added. In order to obtain H3BO3 in the form of light scales a soln. of sp. gr. not exceeding 1.010 (at 90.degree.) was employed. In order to obtain white **crystals** free from coloration all app. should so far as possible be lined with Pb (enameled Fe or earthenware is preferable) and contact with Fe should be avoided; contact with wood colors the soln. yellow. When these precautions were observed perfectly white H3BO3 was produced without the necessity of using the **bone** char required by the Eckelt process. The mother liquors remaining from **crystn**. were used in subsequent operations. The final product was freed from mother liquor and **dried** by centrifuging. Cost data are given; the method is regarded as commercially feasible.

CC 18 (Acids, Alkalis, Salts, and Sundries)

L70 ANSWER 31 OF 37 HCA COPYRIGHT 2003 ACS on STN

13:14963 Original Reference No. 13:2975d-i,2976a-b The cyanides. Commercial sources of ammoniacal nitrogen. Mauge, L. Industrie Chimique (Paris), 5, 286-8 (Unavailable) 1918. CODEN: INCHA8. ISSN: 0537-5606.

AB Cyanides were formerly produced to a large extent by the dry distn. of org. refuse, such as abbatoir waste, and horn, **bone** and leather scrap. More modern processes are the distn. of coal and of waste liquors from beet-sugar refineries. In the purification of coke-oven gas, Fe4(Fe(CN)6)3 and thiocyanates are obtained, which are easily transformable into alkali cyanides. These usually contain an appreciable amt. of cyanate. By the use of a 30% ammonium ferric alum (NH4)4Fe(CN)6 and (NH4)6Fe(Fe(CN)6)2 are obtained in the purification process. These salts are afterwards converted into Ca2Fe(CN)6 by means of CaO and thence into the cyanide. The thiocyanate soln. may be decompd. into NH3 by means of H, CO2, SO3 and H and H2O at high temp. By distn. of beet-sugar waste liquors NH3, CH3OH and N(CH3)3 are obtained. The N(CH3)3 is broken down to HCN and CH4 in another retort at 1000.degree.. The final product contains 5-8% NH3 and 10-12% HCN. The N yield is 25% as NH3, 35% as cyanide and 40% as free N. The saline residue in the retort contains 32% K2CO3, 18% Na2CO3, 20% KCl, 4% **K2SO4** and 25% insol. matter. In the presence of steam, the distn. of beet-sugar liquors yields NH3 directly. (Fr. pat, 442,923, C. A. 7, 2669) HCN can be synthesized in the elec. arc. A mixt. of 70% CH4 and 10% H gives 19% HCN (a yield of 23 g. per kw. hr.). Passing N and moist air over a mixt. of pulverized Al2N3 and C heated to a

high temp. yields HCN (Fr. pat. 435,308). Swan and Kendall use a special gas-heated furnace in which they subject a mixt. of C, N, and alkali to a temp. of 1300.degree.. Very pure cyanide is thereby obtained. Finely powdered Fe is used as a catalyst in another process in which N is caused to react with a mixture of C and alkali or alkaline-earth salts. In this process reaction takes place at a lower temp. and the yield is greater. The mixt. of Fe, C, and Na2CO3 is first heated to the point of fusion and then passed through a die and cut into pieces 6-7 mm. in diam. and 25 mm. long. The fused Fe mixt. is fed into a retort in a gas-heated furnace. Here it reacts with N, produced by passing air through 2 retorts filled with incandescent coke. Na2CO3 + 4C + N2 .fwdarw. 2NaCN + 3CO -138500 cal. Cyanides are converted to NH3 by the action of **hydrated** salts such as K2CO3, Na2CO3, **CaCl2**. The mixt. of cyanides and **hydrate** is introduced into an autoclave along with water and subjected to 12-15 kg. pressure. An almost complete conversion of cyanide N to NH3 is effected. KCN can be converted to NH3 by means of water and steam alone at high temp. (Belgian pat. July 1, 1911). KCN is obtained by allowing air to react with a mixture of coke and K2CO3. The volatile product reacts with the superheated steam introduced into the app. and NH3 is formed. Cyanides (KCN and NaCN) can be transformed into NH3 by heating in an autoclave for 20-30 min. at 180-190.degree. with 5-10 parts of H2O. Formates are obtained as by-products in the reaction and are recovered and converted into HCOOH.

CC 18 (Acids, Alkalis, Salts, and Sundries)

L70 ANSWER 32 OF 37 HCA COPYRIGHT 2003 ACS on STN

13:2792 Original Reference No. 13:491a-i,492a-i,493a-h Rational preparation of superphosphates. Study of the relations connecting the technical process of preparation of superphosphates, chemical constitution and physicomechanical characters of the product. Aita, A. Annali di Chimica Applicata, 10, 45-103 (Unavailable) 1918. CODEN: ACAPAR. ISSN: 0365-1037.

AB As regards the chem. process the industrial prepn. of superphosphates is today still carried out in an empirical direction. Only lately has important work been done in this field (Pratolongo, cf. C. A. 10, 3130; Aita, cf. C. A. 11, 682). Pratolongo asserts that the content in free H3PO4 of superphosphates, besides depending upon the stoichiometrical relations of the reagents, is a close function of the temp. of reaction of formation. A. points out that this statement is clearly in contradiction to the detd. fact in the technical process of superphosphate prepn., i. e., that the use of concns. of **H2SO4** greater than ordinarily employed. effects a remarkable improvement in the physicomechanical state of the product. To clear up this contradiction A. investigated the subject anew in order to verify and control the methods of extn. of free H3PO4 from superphosphates. He then extended his studies further. In the system H3PO4-**CaO**-H2O, which exists essentially in the system H3PO4-**CaO**-H2O-**H2SO4** representing the formation of superphosphate, the following is true: Normally in it

there is a solid phase formed of $CaH_4(PO_4)_2$ and $CaHPO_4$, and a liquid phase composed not only of H_3PO_4 and H_2O but also of $CaH_4(PO_4)_2$; the estn. of the free H_3PO_4 and of the $CaHPO_4$ depends upon the quantity of H_2O that makes a part of the solid phase. Since com. superphosphates run from 12 to 20% in sol. P_2O_5 and from 10 to 20% H_2O, they represent high concns. of P_2O_5 in contact with low quantities of H_2O. On the basis of the statement above, the ideal superphosphate would have a content of free P_2O_5 of from 1/20 to 1/10 of the total sol. P_2O_5. With this in view A. compared the estn. of free P_2O_5 by the various methods available-extn. by 95 % alc., abs. alc., acetone, **anhydrous** Et_2O, H_2O either with direct titration or after addn. of K oxalate. If the solvent neither dissolves not hydrolyzes the $CaH_4(PO_4)_2$, it will ext. from the product not the entire liquid phase but only H_2O and free H_3PO_4. With solvents absolutely **anhydrous** (e. g., Et_2O free of H_2O by distn. over Na) there is not the least trace of **CaO** in the ext. Aq. solvents on the other hand, give exts. containing more or less considerable portions of **CaO**, which varies according to the proportion of H_2O present. Therefore extn. with Et_2O is the only method which furnishes results approximating to the content in free H_3PO_4 theoretically, corresponding to the system H_3PO_4-**CaO**-H_2O. The aq. solvents, owing to their hydrolyzing action upon $CaH_4(PO_4)_2$, ext. from superphosphate appreciably greater fractions of P_2O_5, varying according to the amt. of H_2O they contain. Abs. alc., while containing no H_2O, behaves like an aq. solvent, the presence of the OH group in its structure causing hydrolysis of the $CaH_4(PO_4)_2$. Acetone may serve for extn. of free H_3PO_4 from superphosphate if perfectly pure and **anhydrous**. By the use of the Et_2O extn. method, A. arrived at these conclusions from various expts. on prepns. of superphosphate: (1) Fresh prepns. have a higher content of P_2O_5 than those seasoned; (2) free H_3PO_4 of the liquid phase becomes gradually less and less and tends towards the theoretical amt. in normal superphosphates prepd. by exact stoichiometrical conditions of the reagents; in other words, the conditions altered by the temp. of the reaction during the technical process of prepn. proceed slowly to renew the definite equil. outlined in the system H_3PO_4CaO-H_2O. These considerations (outside of the error due to his method of extn.) explain the increase in content of free H_3PO_4 found by Pratolongo in samples of superphosphate treated in a closed tube in an autoclave at elevated temp. These increases correspond to states of false equil. so that the stable equil. altered by the temp. is not restored to the product except after a relatively long lapse of time. The H_2O contained in superphosphate (using the knowledge of the system H_3PO_4-**CaO**-H_2O as guide) can be looked upon as making up 2 fractions:-H_2O of **crystn**. of the solid constituents, $CaH_4(PO_4)_2$, $CaHPO_4$, **$CaSO_4$**, and humidity, or more properly, the portion of H_2O moistening the product and forming part of the liquid phase. The distinction between these 2 fractions cannot be effected by the ordinary methods of **drying** at different temps. because heating of superphosphates liberates other substances than H_2O. e. g., HF, SiF, besides H_2O of **crystn**

. of some of the salts of the product. Also increase of temp. creates various labile states of equil. in the chem. system out of which the superphosphate is formed, so that there does not correspond to a given temp. any definite state of the system and of the product. Therefore Pratolongo's method of detg. "true humidity" and H2O of **crystn**. is erroneous anti misleading. The use of Et2O instead of EtOH avoids the errors of Pratolongo's method. A.'s method is as follows: Ext. 2 g. of the superphosphate with Et2O, **dry** the fiber (previously tared) together with the residue in an oven at 40.degree., and weigh. The difference this wt. and the orig. 2 g., less the amount of free H3PO4 found in the Et2O ext., is the measure of the H2O of the liquid phase. Keep then in a **desiccator** over 95% **H2SO4** to const. wt.
The difference between the final wt. and the previous wt. gives the amount of H2O of **crystn**. of the solid constituents. A. gives tables of superphosphates derived from various phosphorites, showing total P2O5, P2O5, sol. in H2O and citrate, P2O5 in the form of H3PO4 extd., resp., with Et2O, com. Et2O, abs. alc., and 95 % alc.; H2O of the liquid phase and that of **crystn**., the acid used, the seasoning of the superphosphate, and its physicomechanical properties. A. concludes (1) that the physicomechanical characters of the superphosphate depend on the content of the product in H2O and free H3PO4 taken together. The free P2O5 varies from 0.15 to 9.07%, the H2O of the liquid phase from 1.33 to 15.90% and the H2O of **crystn**. from 1.10 to 4.90%. (2) Free H3PO4 and H2O of superphosphate are intimately bound together, this condition depending particularly upon the concn. of the **H2SO4** employed, on the stoichiometrical conditions of the reagents taking part in the process of formation, but not depending upon the temp. of the reaction. A. divides superphosphates of commerce into 2 classes-normal and abnormal, the first class including those prepd. under exact conditions of stoichiometrical relations between reagents and with not too low concd. acid., the 2nd class including products obtained by defective treatment, either because of excess of **H2SO4**, of low concn. of **H2SO4** or both. J. Kolb (Compt. rend. 78, 825) inferred that the technical reaction of formation of superphosphates was carried out in 2 phases, (a) reaction of 3Ca3(PO4)2 with 6H2SO4 to form 4H3PO4, Ca3(PO4)2 and 6CaSO4; (b) reaction between Ca3(PO4)2 and 4H3PO4 to form 3CaH4(PO4)2. A., from analysis of the mixt. of phosphorite and **H2SO4** at different stages of the reaction, concluded that the Ca3(PO4)2 is attacked exclusively by the **H2SO4** in one reaction with formation of CaH4(PO4)2 and **CaSO4**, with a velocity of reaction depending upon the fineness, nature and content in **CaO** of the phosphorite. In detg. the amount of **H2SO4** to be used to give the largest amount of sol. P2O5 in the superphosphate, the method generally employed is to make repeated tests on new lots of phosphorite on the industrial scale, using varying amounts of **H2SO4**, and checking by analysis of the product. A. believes that equally good or better results can be obtained by the use of empirical factors in connection with the principal components of the phosphorite employed, e. g., Ca3(PO4)2,

$CaCO_3$, Fe_2O_3 and Al_2O_3. The losses sustained by the mixt. of phosphorite and **H_2SO_4** depend largely on the temp. of the reaction. The temp. in turn depends upon the following factors: temp. of the reagents, concn. of the **H_2SO_4**, fineness of the phosphorite meal, nature of the phosphate mineral, and quantity of charge of the reagents. It is most desirable to employ in industrial prepn. the greatest concn. of **H_2SO_4** for the mixt. which is permitted by the app. in use and by reasons of a technical nature. A. considers 3 types of grinding mills: (a) Krupp ball mill, (b) Griffin hammer mill, (c) Kent roller mill. Types (b) and (c) are more recent and are preferred in modern plants to the ball mill because they are easier to handle and give comparatively higher yields for the same power consumption. A. studied the grinding action of (b) on different varieties of phosphate rock, using the Appiani method of levigation to sep. the crushed mineral into 4 fractions, according to the rate of fall through water, i. e., 0.2, 2, 7, 25 mm. per sec. He found that the hard or nodulous varieties (Land Pebble, Angaur) differ from the friable varieties (Gapa, Constantine) in that they give a meal of greater fineness the higher their degree of hardness. The character of the granules of the several varieties also differs. Under the microscope, granules of friable phosphate rock are rounder or with truncated angles, while granules of the hard varieties present sharply angular or flattened forms. The fineness of the meal favors rapid procedure of the reaction of formation, or at least a more complete solubilization of the P_2O_5. Present modes of treatment should not allow over 10-12 % H_2O, nor over 1-2 % of free H_3PO_4, expressed as P_2O_5, corresponding to about 0.05-0.1 of the total sol. P_2O_5. The existence of $CaHPO_4$ is a sure guaranty of the normality of constitution of the product. Normally constituted superphosphate has excellent physicomechanical properties, i. e., **dryness**, pulverulency, abs. absence of pastiness. Abnormally constituted superphosphate has the following characteristics: (1) content in H_2O of the liquid phase above 12%; (2) free H_3PO_4 exceeding the limit of 2% on the wt. of the product, or expressed in P_2O_5, in excess of 0.1 of the sol. P_2O_5; (3) limited amount or absence of $CaHPO_4$; (4) prevalence or abs. presence of the **anhydrous** forms of **crystn.** of the salts $CaH_4(PO_4)_2$, $CaHPO_4$, **$CaSO_4$**; (5) the physicomechanical state of the product is defective and poor, and varies with and depends upon the proportions of H_2O and free H_3PO_4 present, till the product reaches such a degree of pastiness and acidity as to be practically useless and unadapted for spreading by hand or mechanically. A. makes the following suggestions leading to technical prepn. of a good product: (1) The **H_2SO_4** used should have a temp. of 20-30.degree.; (2) the duration of the mixing of the ingredients should not be too long; (3) for hard and nodulous phosphate rock, the concn. of the **H_2SO_4** should be about 54.degree. B.acte.e., and 55-6 for friable phosphate rock, depending upon their degree of humidity; (4) the following factors of parts per part of constituent found in the phosphate rock should be used in calcg. the amount of 53, 54, 55, 56.degree. B.acte.e.**H_2SO_4**, resp., that is to be used:

Ca3(PO4)2, 1.14, 1.11, 1.09, 1 .07; CaCO3, 1.46, 1.43, 1.40, 1.37; Fe2O3(1/2) and Al2O3(1/2) 3.34, 3.25, 3.20, 3.12; not detd., 0.12, 0. 11, 0. 10, 0.09; (5) the weighing should be exactly made, and preferably in the ordinary way, since automatic weighing app. does not always give good results, probably because of the variable pressure exercized upon the columns of phosphate rock meal by different degrees of packing; (6) the charge used in mixing should not contain more than a cwt. of phosphate rock meal, and the time of mixing should be about 15-20 sec.; (7) abnormally constituted superphosphates should not be **dried** out by application of heat. A. recommends the following means as preferable: (a) employment of exhaust fan connected with the reaction chamber, one hr. after completion of charging; (b) **drying** in the cold by use of absorbents such as burnt powdered gypsum or infusorial earth, which are inert, or the use of reacting substances such as powder of **bone** or very friable phosphate rock. These should be sparingly and carefully employed, and their use is at best a mere expedient, in nowise substituting a rational method of prepn. of superphosphate.

CC 15 (Soils, Fertilizers, and Agricultural Poisons)

IT 7664-38-2, Phosphoric acid
(system, **CaO**-H2O-)

IT 7732-18-5, Water
(system, **CaO**-H3PO4-)

L70 ANSWER 33 OF 37 HCA COPYRIGHT 2003 ACS on STN

11:11980 Original Reference No. 11:2458e-i,2459a-e Constitution of internal diazo-oxides (diazophenols). II. Morgan, Gilbert T.; Tomlins, Henry P. (Finsbury Techn. Coll., London). Journal of the Chemical Society, Abstracts, 111, 497-506 (Unavailable) 1917. CODEN: JCSAAZ. ISSN: 0590-9791.

GI For diagram(s), see printed CA Issue.

AB cf. C. A. 9, 2061. The formation of these anhydrides is characteristic of the o- and p-aminophenols, but not of the m-compds. 2,4-H2N(SO3H)C6H3OH (A) was prepd. by the following steps: PhOH .fwdarw. p-HOC6H4SO3H .fwdarw. 2-O2N(HO)C6H3SO3Na .fwdarw. (A). When diazotized by the usual methods it yields the very sol. benzene-2-diazo-1-oxide-4-sulfonic acid (B), HO3SC6H3.O.N2, which, for purposes of isolation, is best prepd. in the absence of non-volatile mineral substances, using purified N2O3 (C. A. 11, 1824). 1 g. finely powdered (A) was suspended in 5 cc. H2O and heated to boiling to dissolve most of the (A). After cooling in a freezing mixt. 2 cc. N2O3 were added, giving a clear, intensely yellow soln. from which (B) sepd. quant. as pale yellow **crystals** with 1 H2O of **crystn**. which is lost at 90.degree. without decompn. of the compd. or change of color. When quickly heated it blackens and decomps. violently 177.degree., but when kept at 115.degree. it suddenly darkens and decomps. with gas evolution. The use of EtONO was unsatisfactory as a substitute for N2O8 but gave good results with "H acid." 4,2-H2N(HO8S)C6H8OH(C) was prepd. by adding p-H2NC6H4OH to 3 parts **H2SO4**, heating 3 hrs. on the H2O bath, adding to H2O, and purifying by **bone**

-blacking the Na salt. Phenol-4-diazonium sulfonate (D) was prepd. from (C) by adding either EtONO or HCl and NaNO2 to a suspension in H2O at 0.degree.. (D), dissolved in C5H5N, gave a yellow, **cryst**. salt which, however, lost all its C5H5N in vacuo over **H2SO4**. No **cryst**. product could be obtained from PhCH2NH2. (D), mixed with excess C5H10NH and placed in a **desiccator** over NaOH-**CaO** to exclude CO2, gave yellow piperidine benzene-4-diazo-1-oxide-2-sulfonate, purified by washing with PhH, turns brownish yellow on **drying** in a **desiccator** and then analyzes for C11H15O4N3S.2/3H2O, has an intense odor like acetamide. A suspension of (D) in cold H2O, treated with excess (PhCH2)2NH, gave dibenzylamine benzene-4-diazo-1-oxide-2-sulfonate, yellow **crystals** with 1 H2O of **crystn**. PhNH2 also gives a yellow salt. All these salts, however, could be at least partially diazoamino compds., but since brucine is a tertiary amine, this objection could not apply to the brucine salt, from brucine HCl and (D) in H2O, followed by 1 equiv. of Na2CO8, bright yellow leaflets with 1 H2O; formulas (I) or (II) are assigned. Metallic salts were not isolated. At room temp. in the presence of excess NH3 (D) gives off its diazo N only very slowly, 88% being eliminated after 8 days, and very little tendency for azo compd. formation being shown. m-H2NC6H4OH was sulfonated as in the case of the p-compd., the acid purified by **recrystn**. from H2O, and diazotized in the form of a finely divided suspension obtained by acidifying a soln. of the Na salt with HCl. The resulting phenol-3-diazonium-4-sulfonate (E), HOC6H3.SO2.O.N2, forms a yellowish white ppt. which decomps. at 86.degree. with effervescence, contains H2O of **crystn**., and loses N even at room temp. An attempt to prep. the brucine salt failed, only a few orange-colored **crystals** being obtained. The orange color is due to the very sol. dye (III), which forms when (E) is treated with excess NH3, only 0.5 the diazo N being evolved.

CC 10 (Organic Chemistry)

L70 ANSWER 34 OF 37 HCA COPYRIGHT 2003 ACS on STN

10:17090 Original Reference No. 10:3130f-i,3131a-d Technical chemical researches on superphosphates. Pratolongo, Ugo Annali di Chimica Applicata, 6, 59-112 (Unavailable) 1916. CODEN: ACAPAR. ISSN: 0365-1037.

AB In the technical prepn. of superphosphates, the conditions that influence the degree of pulverulency and of **dryness** of the product are very important, as these characters have a direct relation to the efficient use of superphosphates in agrarian practice. **Drying** of the product is ordinarily brought about by moderate heating or by successive additions of phosphorite lime, gypsum or other inert substance. The chem. compn. and the physical characteristics of superphosphates depend upon the proportion of **H2SO4** used and the thermal behavior of the reactions. **CaSO4** occurs in mineral phosphates in the 2 forms, gypsum (**CaSO4**.2H2O) and anhydrite,. **CaSO4** , usually as anhydrite. When both forms occur together, anhydrite is nearly always the dominant form. In **bone**

superphosphates as generally prepd. with limited amt. of **H2SO4**, **CaSO4** assumes more frequently the form of gypsum. Mono-Ca phosphate is commonly the monohydrate, but occasionally is **anhydrous**; the same may be said of di-Ca phosphate. In studying the constitution of superphosphates, P. detd. the "true humidity" (moisture sol. in alc.), the H2O of **crystn.**, total H2O (loss on heating 4 hrs. at 130.degree.) free H3PO4 (by titration of alc. ext.) and total P2O5. Many analyses were made. Free H3PO4 ranged from 3.7% to 19.9%. In the constitution of superphosphates, a liquid phase participates. This phase is essentially an aq. soln. of H3PO4 and **CaO**. Increasing amts. of free H3PO4 and total H2O bring about a decreasing degree of **dryness** and pulverulency of the product. Products in which the liquid phase (true humidity + free H3PO4) is less than 15% are very **dry** and pulverulent, from 15 to 18% fair, from 18 to 25% somewhat wet to the touch, and above 25% wet, lumpy and easily compressible. In the technical preparation of superphosphates, the state of **hydration** in which the mono- and di-Ca phosphates sep. out in the reaction, and the content of the product in free H3PO4 (i. e., the amount and compn. of the liquid phase) are functions, at the same time, of the temp. or rather thermal procedure of the reaction, and of the diln. of the **H2SO4** employed. Commonly **CaSO4** seps. at first as anhydrite and rarely also as the semihydrate, 2CaSO4.H2O. The gypsum that often occurs in superphosphates must be considered as derived secondarily by successive **hydrations** of anhydrite or the semihydrate. Mono- and di-Ca phosphates sep. out as **anhydrous** or **hydrated** forms according to the temp. at which the sepn. takes place and according to the compn. of the liquid phase from which they sep. out. On these 2 factors depends the character of the greater or less pulverulency of superphosphates. In order to arrive at technical success in manufacturing **dry** and pulverulent superphosphates, the following conditions must be studied and fixed, as they det. the thermal procedure of the process: initial temp. of the reagents, amount and concn. of the **H2SO4**, degree of fineness of the phosphorite, capacity of the mechanical mixer, time of kneading, and aeration of the kneading chamber.

CC 15 (Soils and Fertilizers)

L70 ANSWER 35 OF 37 HCA

3:10521 Original Reference No. 3:1941e-i Occurrence of Gluconic Acid in an Efflorescence from the Walls of a Sugar Storehouse. Stanek, Vladimir (Prague Sugar Experiment Sta.). Zeitschrift fuer Zuckerindustrie in Boehmen, 33, 547-51 (Unavailable) 1909. CODEN: ZZIBAJ. ISSN: 0373-0409.

AB In a sugar storehouse at Nimburg, a brownish colored efflorescence permeated with mould was found upon the stone walls behind the bagging material used for covering the same. Analysis of the efflorescence gave the following: H2O 13.60%, organic substance insol. in H2O 5.15%, sand 3.25%, Fe2O3 and Al2O3 0.25%, **CaO** 8.35%, MgO 0.22%, alkalies 2.50%, organic substances sol. in H2O

66.68%. 1 kg. of the material **recrystallized**, using **bone**-black, gave 450 g. of a colorless finely **crystalline** Ca organic salt. Upon **recryst**. the product gave 12.34-12.47% **CaO**. The **crystals** were easily sol. in H2O and insol. in alc. 5 g. substance in 100 cc. gave a rotation of + 2.3 .degree. in the saccharimeter. 150 g. salt were decomposed with the equivalent amt. of **H2SO4**. The **CaSO4** was filtered off, the filtrate evaporated in vacuum and the dissolved **CaSO4** pptd. out with 3 vols. of alc. The alc. sol. was evaporated on the water bath and after several days' standing in the **desiccator crystallized** to a mass of finely pointed needles. Fractions of the compound obtained melted at 132.degree., was easily sol. in H2O less sol. in alc., had a sweet taste, did not reduce Fehling sol. and gave no ppt. with Pb subacetate. 2 g. substance in 100 cc. polarized after 30 sec. + 7.0.degree., after 5 min. + 7.degree., after 20 hrs. + 6.1.degree., after 68 hrs. + 5.3.degree.. Elementary analyses gave results corresponding to the formula C6H10O8, all of which characteristics coincide with those of the lactone of gluconic acid. Analyses of the Zn salt furnished additional identification. The formation of the gluconic acid is explained by the absorption of the sirup from the stored sugar by the bag covering upon the walls, the inversion and oxidation of this sugar to gluconic acid by microorganisms and the conversion of the gluconic acid to the Ca salt upon the stone walls of the storehouse.

CC 28 (Sugar, Starch, and Gums)

L70 ANSWER 36 OF 37 HCA COPYRIGHT 2003 ACS on STN

3:8957 Original Reference No. 3:1667f-i,1668a Determination of Malic Acid in Wine. von der Heide, C.; Steiner, H. (Oenochem. Versuchst. Geisenheim.). Zeitschrift fuer Untersuchung der Nahrungs- und Genussmittel sowie der Gebrauchsgegenstaende, 17, 307 (Unavailable) 1909. CODEN: ZNGEA2. ISSN: 0372-9419.

AB The authors determine the combined amount of malic and succinic acid and then deduct from this the amount of succinic acid obtained by another method (see preceding abst.). Malic and succinic acid together are determined as follows: 50 cc. of wine in a beaker are treated with 1 cc. glac. AcOH, 0.25 cc. 20% KOAc and 7.5 g. **CaCl2**. After solution of the last, 7.5 cc. of 95% alc. are added. After stirring, the mixture is allowed to stand for 15 min., filtered and washed with not more than 10 cc. of a solution of 15 g. **CaCl2**, 20 cc. 95% alc. and 100 cc. water. The filtrate is evaporated to a few cc. until the HOAc is driven off, breaking the crust of **crystals** with a pestle. Take up in a little water, add 5 cc. 10% BaCl2 and powd. Ba(OH)2 until faintly alkaline to phenolphthalein. Remove excess of Ba(OH)2 by passing CO2 over the surface while stirring. Cool, bring the vol. to exactly 20 cc. and add 85 cc. 96% alc. After 2 hrs. filter, and wash the ppt. with 80% alc. Wash the ppt. back into the dish with hot water and evaporate almost to dryness, breaking the crust of **crystals** with a pestle. Moisten the residue with 2.5-3 cc. 40% H2SO4 and slowly add anhyd. **Na2SO4**, triturating with a pestle until

the whole forms a light dry powder. Extract this in a Soxhlet apparatus with ether for 6 hrs. Add 10-20 cc. water to the ether extract, evaporate the ether, decolorize the residue with 1-3 g. purified **bone** char, filter, wash, neutralize the filtrate with 0.1 N alkali, evaporate to dryness on a water bath, ash at a low red heat, and titrate the alkali carbonate in the ash. With the amount of succinic acid known, the titer due to carbonate from this may be deducted from that of the total carbonate, the balance being calculated to malic acid. A series of control analyses upon artificial wines of known malic acid content resulted in the recovery of 96.3-103.2% of the acid added.

CC 16 (Fermented and Distilled Liquors)

L70 ANSWER 37 OF 37 HCA

3:1304 Original Reference No. 3:253f-i,254a-i,255a-d Analytical Methods in the Manufacture of Stearin. Freundlich, J. Chemische Revue ueber die Fett- und Harz-Industrie, 15, 224-7,246-9,277-8 (Unavailable) 1909. CODEN: CRFHAJ. ISSN: 0366-7960.

AB A description of the methods used in manufacturing stearin by the following method: Raw materials-tallow, palm oil and **bone** fat. Method of saponification-a theoretically insufficient amount of MgO in the autoclave at a pressure of 8 to 10 atm. Further treatment-drawing off the glycerol water, decomp. of fatty mass by dil. **H2SO4**, washing, **drying** and vacuum distillation, cold pressing, second distillation, and warm-pressing. ANALYTICAL METHODS. 1. Fats: Sampling of tallow and **bone** fat is done by long lard trier from as many tierces as possible; of palm oil by emptying tierces into a reservoir, mixing all at temp. near solidifying point to a homogeneous mass and then drawing samples from different parts of the reservoir. (a) Tallow: Moisture is determined by **drying** at 100-10.degree. to constant weight. Ash by careful burning and ashing of a weighed amount. The free fatty acids are calculated from the following formula assuming that the mean mol. wt. of the free and combined acids are alike: f = (168300-38d)S/1683K. f= % free fatty acids, S = acid number, k = sapon. number, d = k - s or ester number. The yield of glycerol is calculated from the formula: G = 92d/3 .times. 561 = 0.05466d, G being % of glycerol and d being the ester number. The titer of the fatty acids is determined by the well-known Dalican method. Oleic acid is calculated from the I figure, assuming that pure oleic acid absorbs 90.970% I; O = I/90.07 .times. 100, O being % oleic acid and I the I number. The Hehner value as ordinarily determined must be corrected for unsaponifiable matter if such should be present. The unsaponifiable matter is calculated from the formula: U = 100 - 100s1/s2, where s is the neutralization number of the total "Hehner" fatty acids before removing the unsaponifiable matter and s2 the neutralization number after removal. (b) Palm oil: The moisture det. by **drying** at 100-10.degree. gives slightly high results on account of the volatility of free glycerol. Organic impurities are determined by extracting the **dried** sample with ether, weighing the residue and subtracting from it its mineral matter by ashing residue. The ash, free fatty acids and titer are

determined as for tallow. Glycerol: a weighed amount is saponified, decomposed with **H2SO4**, the filtrate is neutralized with BaCO3, filtrate from it is concentrated, extracted with ether-alcohol, evaporated off and residue subjected to acetin method for glycerol. The Hehner value is determined as usual, but to remove the impurities the aqueous solution of the soap must be filtered and is then decomposed by acid. (c) **Bone** fat: On account of the high ash which causes emulsions in the settling from the autoclave treatment, it is essential to boil the **bone** fat first with dilute **H2SO4**. To calculate the amount of acid necessary the ash is figured as **CaO** and this to **H2SO4** and an excess added. Before filling the autoclave the acid must be removed from the **bone** fat by thorough washing. All determinations are made as directed for tallow. 2. Magnesia: Determinations are made for loss on ignition; total alkalinity by titration with excess of standard acid and re-titration by alkali; CO2 by absorption in KOH; also SiO2 and **CaO**; the latter decreases the value of MgO by formation of **CaSO4** during the acid treatment. 3. Sulphuric Acid: Its strength is determined by the usual methods of inorganic analysis. 4. Autoclave Samples: Samples are drawn by means of a thin tube which is mounted in the cover of the autoclave and reaches to the center of same; by opening the valve at the upper end of the tube the pressure in the autoclave forces the contents up and out through the tube. The sample which consists of free fatty acids, neutral fat and magnesia soap is boiled with dilute **H2SO4**, washed, filtered, **dried** and tested for free fatty acids. Assuming that the sapon. number is 200, then half of the acid number equals approx. the percentage of free fatty acids. 5. Magnesia Soap from Settling Basin after Autoclave Treatment: The sample is tested to find the proper amount of **H2SO4** necessary for decomposing the soap. This may also be calculated from the original ash of the raw fat plus the MgO which had been added. 6. Decomposed Crude Fatty Acids: The crude fatty acids are tested for ash contents which should be zero and must not exceed a few hundredths of 1%, otherwise foaming and bumping will occur during distillation. If more ash is found than permissible, the decomp. with **H2SO4** is repeated. The sample is also tested for SO3 by boiling a quantity with an equal volume of water, acidifying with HCl and adding BaCl2; no turbidity should occur. 7. Acid Water: Tested for acidity to prevent excessive use of **H2SO4**. 8. Glycerol Water: Tested by hydrometer usually. This test indicates any irregularity in the process. If desired the glycerol may be concentrated by evaporation at 80.degree. and its percentage determined by the acetin method. 9. Crude Glycerol: Tested by Westphal balance or by the acetin method. The ash determination is also valuable; a high ash lowers the quality and quantity of the distillate. 10. Tar: About 10 g. are dissolved in neutralized ether; the pitch and asphalt is precipitated by neutralized abs. alcohol (ratio of alcohol: ether = 4: 3) and allowed to settle twelve hours. Filter and determine free fatty acids with 0. 1 N alcoholic NaOH. Evaporate the neutralized solution, take up residue with a little benzene, saponify with 0.5 N

NaOH and titrate back with HCl; calculate the neutral fat from the 0.5 N NaOH consumed. 11. Stearin, Once Distilled: Determine titer, I number and acid figure; the last should be higher than in 6. 12. The "stearin" which is obtained by cooling the crude olein is tested for neutral fat, titer and I number. 13. Fatty Acids Once Distilled and Cold-pressed: Test for titer and I number; the former should be higher, the latter lower than in 11. 14. Fatty Acids, Twice Distilled Once Cold-pressed: Determine titer and I number. Palmitic acid has the lower b. p., stearic acid the higher b. p. If any acrid acrolein odor is perceptible, test for unsaponifiable matter and for neutral fat. 15. In the return olein liquors determine the titer and I number. 16. Olein: Determine the I number; the purer the olein the higher will be this number. Neutral fat is present if during distillation the mass should foam over; this is usually shown by a dark color of the distillate. 17. Distilled Olein: Same as 16. 18. Prime Stearin: Generally only palmitic, stearic and oleic acids are present; iso-oleic acid, stearolactone and hydroxystearic acid are present only when saponification with **H2SO4** is practiced. Iso-oleic acid increases the I number. Stearolactone is calculated from the difference between the neutralization figure and sapon. number; if this difference is zero, stearolactone is absent. If oleic acid is known (from I number) the palmitic and stearic acids are calculated from the following two equations: $X + y + O = 100$; $x/284 + y/256 + O/282 = 100/M$; where x equals % stearic acid, y equals % palmitic acid, O equals % oleic acid and M equals mean mol. wt. of the total fatty acids. 19. Stearin Thrice Distilled, Once Cold-pressed: Determine I number and titer. 20. Goudron, Tarry Residue: Determine the unsaponifiable matter and the neutral fat as in 10. 21. Distilled Glycerol: Determine sp. gr. by the Westphal balance or pycnometer and the ash; the latter should not be above 0.2%. The author emphasizes also the importance of an exact account of raw materials used, of yield of products and of a record of monthly samples and their analysis.

CC 27 (Fats, Fatty Oils, and Soap)

=> d l71 1-23 cbib abs hitstr hitind

L71 ANSWER 1 OF 23 HCA COPYRIGHT 2003 ACS on STN

138:276344 Pore-forming agents for orthopedic cements. Dalal, Paresh S.; Landeryou, Tracy J.; Toth, Carol Ann; Kulkarni, Shailesh C. (Stryker Corporation, USA). PCT Int. Appl. WO 2003024316 A2 20030327, 106 pp. DESIGNATED STATES: W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, BF, BJ, CF, CG, CH, CI, CM, CY, DE, DK, ES, FI, FR, GA, GB, GR, IE, IT, LU, MC, ML, MR, NE, NL, PT, SE, SN, TD, TG, TR. (English). CODEN: PIXXD2. APPLICATION: WO 2002-US29966 20020920. PRIORITY: US 2001-960421

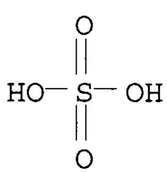
20010921.

AB A **bone** precursor compn. comprising a cement mixt. and a pore-forming agent is provided for **bone** implant. Preferably, the pore-forming agent has a particle size of 20-500 .mu.m. More preferably, the proportion of the pore-forming agent is 7-40% (wt./wt.). The compn. may further include a bioactive agent, preferably a **bone** morphogenetic protein or nucleic acid encoding BMP encapsulated in the pore-forming agent. The moldability of the compn. can be modulated by the addn. of a binder. The invention provides a kit and implant device comprising the **bone** precursor compn. The invention also provides an implantable prosthetic device comprising a prosthetic implant having a surface region and a **bone** precursor material disposed on the surface region. The kit and devices may further comprise one or more addnl. components including a bioactive agent and a binder. Hydroxyapatite implants were prepd. contg. PLGA microspheres and pore-forming agents such as Ca sulfate, tetracalcium phosphate and dicalcium phosphate.

IT **7778-18-9**, Calcium sulfate **10101-41-4**, **Calcium sulfate dihydrate**
(pore-forming agents for orthopedic cements)

RN 7778-18-9 HCA

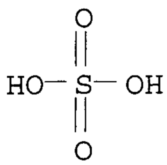
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

RN 10101-41-4 HCA

CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



Ca

2 H_2O

IC ICM A61B
CC 63-7 (Pharmaceuticals)
IT **Bone** morphogenetic proteins
(10; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(12; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(13; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(14; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(15; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(16; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(17; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(18; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(2; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(3; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(4; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(5; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(6; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(7; pore-forming agents for orthopedic cements)
IT **Bone** morphogenetic proteins
(9; pore-forming agents for orthopedic cements)
IT Medical goods
(**bone** cements; pore-forming agents for orthopedic

cements)

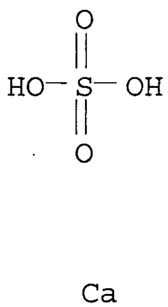
IT 471-34-1, Calcium carbonate, biological studies 1306-01-0, Tetracalcium phosphate 1306-06-5, Hydroxyapatite 7757-93-9, Dicalcium phosphate 7758-87-4, Tricalcium phosphate **7778-18-9**, Calcium sulfate 7789-77-7, Dicalcium phosphate dihydrate 9003-01-4, Polyacrylic acid 10031-30-8, Monocalcium phosphate monohydrate 10034-76-1, Calcium sulfate hemihydrate 10086-45-0, Calcium pyrophosphate **10101-41-4**, **Calcium sulfate dihydrate** 14096-86-7 24937-78-8, Eva 24980-41-4, Polycaprolactone 25248-42-4, Polycaprolactone 26009-03-0, Polyglycolide 26023-30-3, Poly[oxy(1-methyl-2-oxo-1,2-ethanediyl)] 26161-42-2 26202-08-4, Polyglycolide 26680-10-4, Polylactide 26780-50-7, Polyglactin 27083-66-5, Polypropylene fumarate 28728-97-4, Poly[oxy(1-oxo-1,4-butanediyl)] 29223-92-5, 1,4-Dioxan-2-one, homopolymer 31621-87-1, Polydioxanone 31852-84-3, Poly(trimethylene carbonate) 33135-50-1, Poly(L-lactide) 41706-81-4, .epsilon.-Caprolactone-glycolide copolymer 50862-75-4, Poly(oxycarbonyloxy-1,3-propanediyl) 52352-27-9, Poly(hydroxybutyric acid) 53801-86-8, Calcium metaphosphate 75734-93-9, Glycolide-trimethylene carbonate copolymer 129515-24-8, Lactide-trimethylene carbonate copolymer 193830-08-9, Gdf-5 193830-09-0, Gdf-6 193830-10-3, Gdf-7 208778-50-1, Gdf-9 252671-88-8 252959-51-6, **Bone** morphogenetic protein 11 271597-10-5, Gdf-1 271597-11-6, Gdf-3 271597-12-7, Gdf-8 271597-13-8, **Bone** morphogenetic protein 3b 271597-14-9, Gdf-12 305835-60-3, Gdf-2
(pore-forming agents for orthopedic cements)

L71 ANSWER 2 OF 23 HCA COPYRIGHT 2003 ACS on STN

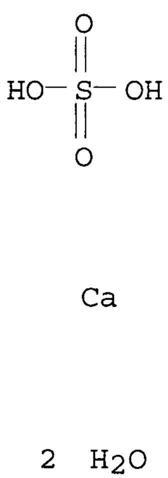
138:158909 Time release calcium sulfate matrix for **bone** augmentation. Ricci, John L.; Alexander, Harold; Hollander, Bruce L. (Biolok International, Inc., USA). PCT Int. Appl. WO 2003011957 A1 20030213, 31 pp. DESIGNATED STATES: W: AE, AT, AU, AZ, BG, BR, BY, CA, CH, CN, CR, CU, DE, EE, ES, FI, GB, GE, HR, HU, ID, IL, IN, JP, KG, KR, KZ, LT, LV, MA, MD, MK, MX, MZ, NO, NZ, PL, PT, RO, RU, SE, SG, SI, SK, TJ, TM, TR, TT, UA, US, UZ, YU, ZA, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR. (English). CODEN: PIXXD2. APPLICATION: WO 2002-US23981 20020726. PRIORITY: US 2001-918445 20010801.

AB An implant compn. having controlled resorption rate in vivo for stimulating **bone** growth, a method of making the implant compn., and a kit of implant materials are disclosed. The implant compn. includes a calcium sulfate compd., polymer contg. particles, and a setting agent for setting the calcium sulfate compd. and the polymer contg. particles into a heterogeneous solid compn. Upon setting, the calcium sulfate compd. forms a matrix and the polymer contg. particles settled within the matrix. The resorption rate of the implant compn. in vivo can be controlled of between eight and 24 wk, which substantially matches the rate of **bone** growth.
The implant compn. can be used for the repair, augmentation, and

other treatment of **bone**.
IT **7778-18-9**, Calcium sulfate **10101-41-4**,
Calcium sulfate dihydrate
(time release **calcium sulfate** matrix for
bone augmentation)
RN 7778-18-9 HCA
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



RN 10101-41-4 HCA
CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



IC ICM C08K003-30
ICS C08K009-00; A61K006-08; A61K006-087
CC 63-7 (Pharmaceuticals)
ST time release calcium sulfate matrix **bone** implant
IT Polyesters, biological studies
(caprolactone-based; time release calcium sulfate matrix for
bone augmentation)
IT Prosthetic materials and Prosthetics
(implants; time release calcium sulfate matrix for **bone**
augmentation)
IT Polyesters, biological studies

(lactide; time release calcium sulfate matrix for **bone** augmentation)

IT **Bone**
(resorption; time release calcium sulfate matrix for **bone** augmentation)

IT **Bone**
Coating materials
(time release calcium sulfate matrix for **bone** augmentation)

IT Alkali metal salts
(time release calcium sulfate matrix for **bone** augmentation)

IT Carnauba wax
Polyesters, biological studies
Polymers, biological studies
(time release calcium sulfate matrix for **bone** augmentation)

IT **7778-18-9**, Calcium sulfate 9002-89-5, Poly(vinyl alcohol) 10034-76-1, Calcium sulfate hemihydrate **10101-41-4**, **Calcium sulfate dihydrate** 24980-41-4, Poly(.epsilon.-caprolactone) 25248-42-4, Poly[oxy(1-oxo-1,6-hexanediyl)] 26009-03-0, Polyglycolide 26023-30-3, Poly[oxy(1-methyl-2-oxo-1,2-ethanediyl)] 26202-08-4, Polyglycolide 26680-10-4, Polylactide 29223-92-5 31621-87-1, Polydioxanone
(time release **calcium sulfate** matrix for **bone** augmentation)

L71 ANSWER 3 OF 23 HCA COPYRIGHT 2003 ACS on STN

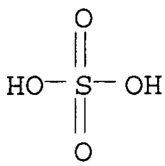
137:375322 Orthopedic **bone** filling materials containing calcium sulfate pastes with good workability. Hayashi, Tomokazu; Hayashi, Kiyotomi (Japan). Jpn. Kokai Tokkyo Koho JP 2002331026 A2 20021119, 5 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2001-127868 20010425.

AB The filling materials, which are applied to deformed **bone** such as cavity of **bone** or vertebra and finally absorbed by the tissue, contain CaSO4-contg. paste with viscosity 20-75 cPs, preferably 30-60 cPs. **CaSO4**.cntdot.1/**2H2O** (10 g) was kneaded with 3.6 mL H2O at 27.degree. and 30 rpm for 1 min and kept for 5 min. The paste with viscosity 30 cPs thus obtained was easily ejected into saline and hardened after 20 min, while a control paste with 67 cPs obtained by keeping for 13 min before ejection was slightly hard to be ejected.

IT **7778-18-9**, Calcium sulfate
(orthopedic **bone** filling materials contg. CaSO4 paste with controlled viscosity for good workability)

RN 7778-18-9 HCA

CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

IC ICM A61L027-00
CC 63-7 (Pharmaceuticals)
ST **bone** filling viscosity controlled calcium sulfate paste; deformed **bone** restoration calcium sulfate paste
IT Medical goods
(biodegradable; orthopedic **bone** filling materials contg. CaSO4 paste with controlled viscosity for good workability)
IT Medical goods
(**bone** cements; orthopedic **bone** filling materials contg. CaSO4 paste with controlled viscosity for good workability)
IT **Bone**
(deformation, restoration of; orthopedic **bone** filling materials contg. CaSO4 paste with controlled viscosity for good workability)
IT Biodegradable materials
(medical; orthopedic **bone** filling materials contg. CaSO4 paste with controlled viscosity for good workability)
IT **7778-18-9**, Calcium sulfate 10034-76-1, Calcium sulfate hemihydrate
(orthopedic **bone** filling materials contg. CaSO4 paste with controlled viscosity for good workability)

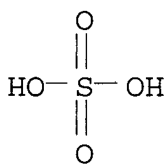
L71 ANSWER 4 OF 23 HCA COPYRIGHT 2003 ACS on STN
137:342179 Calcium phosphate/sulphate-based **bone** implant composition. Cooper, John Joseph (Biocomposites Limited, UK). PCT Int. Appl. WO 2002087649 A1 20021107, 16 pp. DESIGNATED STATES: W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, BF, BJ, CF, CG, CH, CI, CM, CY, DE, DK, ES, FI, FR, GA, GB, GR, IE, IT, LU, MC, ML, MR, NE, NL, PT, SE, SN, TD, TG, TR. (English). CODEN: PIXXD2. APPLICATION: WO 2002-GB1986 20020501. PRIORITY: GB 2001-10726 20010502.
AB A **bone** implant compn., the compn. comprising calcium

sulfate and slowly sol. sources of calcium, orthophosphate and hydroxyl ions. The compn. may be provided in powder or granulated form. A powd. mixt. was prepd. from .beta.-tricalcium phosphate 1.25, calcium sulfate .alpha.-hemihydrate 0.63, and MgO 0.05 g. The .beta.-tricalcium phosphate particles have a size of 250-500 .mu.. The mixt. was blended with 0.85 mL 1% potassium sulfate soln. to give a paste which was used to fill a periodontal pocket.

IT **7778-18-9**, Calcium sulfate **10101-41-4**, **Calcium sulfate dihydrate**
(**calcium** phosphate/**sulfate**-based **bone** implant compn.)

RN 7778-18-9 HCA

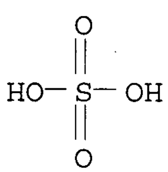
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

RN 10101-41-4 HCA

CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



Ca

2 H_2O

IC ICM A61L027-42
ICS A61C008-00; A61F002-28; A61F002-46; A61L024-00

CC 63-7 (Pharmaceuticals)

ST calcium phosphate sulfate **bone** implant

IT **Bone**

(artificial; calcium phosphate/sulfate-based **bone** implant compn.)

IT Antibiotics
Antitumor agents
Dental materials and appliances
Particle size distribution
(calcium phosphate/sulfate-based **bone** implant compn.)

IT **Bone** morphogenetic proteins
(calcium phosphate/sulfate-based **bone** implant compn.)

IT Drug delivery systems
(granules; calcium phosphate/sulfate-based **bone** implant compn.)

IT Drug delivery systems
Prosthetic materials and Prosthetics
(implants; calcium phosphate/sulfate-based **bone** implant compn.)

IT Drug delivery systems
(pellets; calcium phosphate/sulfate-based **bone** implant compn.)

IT 471-34-1, Calcium carbonate, biological studies 546-93-0D, basic 1305-62-0, Calcium hydroxide, biological studies 1305-78-8, Calcium oxide, biological studies 1306-01-0, Tetracalcium phosphate 1306-06-5, Hydroxylapatite 1309-48-4, Magnesium oxide (MgO), biological studies 1314-13-2, Zinc oxide (ZnO), biological studies 1403-66-3, Gentamicin 1592-23-0, Calcium stearate 7440-70-2, Calcium, biological studies 7693-13-2, Calcium citrate 7757-93-9, Dicalcium phosphate 7758-87-4, Tricalcium phosphate **7778-18-9**, Calcium sulfate 7789-75-5, Calcium fluoride (CaF2), biological studies 10034-76-1, Calcium sulfate hemihydrate 10043-83-1, Magnesium orthophosphate **10101-41-4**, **Calcium sulfate dihydrate** 10103-46-5, Calcium phosphate 16389-88-1, Dolomite, biological studies 20427-58-1, Zinc hydroxide (Zn(OH)2)
(**calcium** phosphate/**sulfate**-based **bone** implant compn.)

L71 ANSWER 5 OF 23 HCA COPYRIGHT 2003 ACS on STN

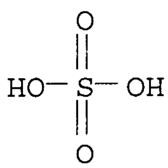
137:52426 Osteological material and its usage. Lin, Zhiyi; Lin, Shengfu (Peop. Rep. China). Faming Zhuanli Shenqing Gongkai Shuomingshu CN 1306865 A 20010808, 7 pp. (Chinese). CODEN: CNXXEV. APPLICATION: CN 2000-100673 20000127.

AB A process comprises mixing **CaSO4** 1/**2H2O** with water at a ratio of 15-80:85-20, homogenizing to viscosity of 20-75 ps, and filling in **bone** skeleton or vertebra.

IT **7778-18-9**, Calcium sulfate
(calcium sulfate **bone** prosthetic)

RN 7778-18-9 HCA

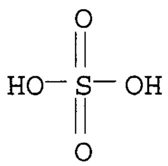
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

IC ICM A61L031-02
ICS A61L024-02
CC 63-7 (Pharmaceuticals)
ST **bone** prosthetic material calcium sulfate
IT **Bone**
(artificial; calcium sulfate **bone** prosthetic)
IT Prosthetic materials and Prosthetics
(calcium sulfate **bone** prosthetic)
IT **7778-18-9**, Calcium sulfate
(calcium sulfate **bone** prosthetic)

L71 ANSWER 6 OF 23 HCA COPYRIGHT 2003 ACS on STN
136:11065 New pharmaceutical composition. Papadimitriou, Apollon (F. Hoffmann-La Roche A.-G., Switz.). PCT Int. Appl. WO 2001087329 A1 20011122, 64 pp. DESIGNATED STATES: W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CO, CU, CZ, DE, DK, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, BF, BJ, CF, CG, CH, CI, CM, CY, DE, DK, ES, FI, FR, GA, GB, GR, IE, IT, LU, MC, ML, MR, NE, NL, PT, SE, SN, TD, TG, TR. (English). CODEN: PIXXD2. APPLICATION: WO 2001-EP5187 20010508. PRIORITY: EP 2000-110355 20000515.
AB The present invention relates to a liq. pharmaceutical compn. comprising an erythropoietin protein, a multiple charged inorg. anion in a pharmaceutically acceptable buffer suitable to keep the soln. pH in the range from about 5.5 to about 7.0, and optionally one or more pharmaceutically acceptable excipients. This compn. is esp. useful for the prophylaxis and treatment of diseases related to erythropoiesis.
IT **7757-82-6**, Sodium sulfate, uses
(buffer; stabilized erythropoietin pharmaceutical compn.)
RN 7757-82-6 HCA
CN Sulfuric acid disodium salt (8CI, 9CI) (CA INDEX NAME)



2 Na

IT **10043-52-4**, Calcium chloride, biological studies
(stabilized erythropoietin pharmaceutical compn.)
RN 10043-52-4 HCA
CN Calcium chloride (CaCl2) (9CI) (CA INDEX NAME)

Cl—Ca—Cl

IC ICM A61K038-18
ICS A61K009-08; A61K047-02; A61K047-18
CC 63-3 (Pharmaceuticals)
Section cross-reference(s): 2, 16
IT Drug delivery systems
(freeze-**dried**; stabilized erythropoietin pharmaceutical compn.)
IT **Drying**
(spray; stabilized erythropoietin pharmaceutical compn.)
IT Anemia (disease)
Antioxidants
Bone marrow
Buffers
Electrophoresis
Erythrocyte
Erythropoiesis
Fermentation
Molecular cloning
Preparative chromatography
Preservatives
Protein sequences
Reticulocyte
pH
(stabilized erythropoietin pharmaceutical compn.)
IT 74-79-3, Arginine, uses 7664-93-9, Sulfuric acid, uses
7757-82-6, Sodium sulfate, uses
(buffer; stabilized erythropoietin pharmaceutical compn.)
IT 50-70-4, Sorbitol, biological studies 56-81-5, Glycerol, biological studies 57-50-1, Saccharose, biological studies 63-68-3, Methionine, biological studies 69-65-8, Mannitol 99-20-7, Trehalose 9005-64-5, Polysorbate 20 9005-65-6,

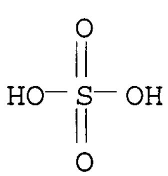
Polysorbate 80 **10043-52-4**, Calcium chloride, biological studies 25322-68-3D, Polyethylene glycol, protein conjugates 106392-12-5, Pluronic f68
(stabilized erythropoietin pharmaceutical compn.)

L71 ANSWER 7 OF 23 HCA COPYRIGHT 2003 ACS on STN
134:316186 Implantation of surgical implants with calcium sulfate. Ricci, John; Alexander, Harold; Berman, Charles L.; Frenkel, Sally; Hollander, Bruce; Pecora, Gabriele (Hospital for Joint Diseases, USA). U.S. US 6224635 B1 20010501, 7 pp. (English). CODEN: USXXAM. APPLICATION: US 1998-187584 19981106.

AB The present invention relates to techniques for the prepn. and implantation of implants with surgical cements composed primarily of calcium sulfate ("CS"). The first of these novel techniques involves the steps of: (1.) precoating an implant with CS; (2.) permitting the implant to dry, and, (3.) thereafter grouting the implant in place with wet CS. The second embodiment involves: (1.) grouting an uncoated implant in place with wet CS. Finally, the third embodiment involves: (1.) precoating an implant with CS; (2.) permitting it to dry; and, (2.) subsequently press-fitting the implant in place without grouting.

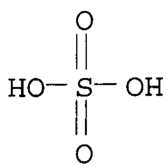
IT **7778-18-9**, Calcium sulfate **10101-41-4**, **Calcium sulfate dihydrate**
(implantation of surgical implants with calcium sulfate)

RN 7778-18-9 HCA
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

RN 10101-41-4 HCA
CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)

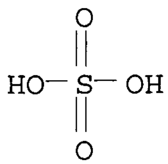


Ca

2 H_2O

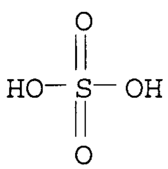
IC ICM A61K006-08
ICS A61F002-28
NCL 623023620
CC 63-7 (Pharmaceuticals)
IT Medical goods
(**bone** cements; implantation of surgical implants with calcium sulfate)
IT **7778-18-9**, Calcium sulfate 10034-76-1, Calcium sulfate hemihydrate **10101-41-4**, **Calcium sulfate dihydrate**
(implantation of surgical implants with calcium sulfate)

L71 ANSWER 8 OF 23 HCA
134:183552 **Bone** substitute for implantation in the human and animal body. Roessler, Ralf (Ivoclar Vivadent A.-G., Liechtenstein). PCT Int. Appl. WO 2001012242 A1 20010222, 27 pp. DESIGNATED STATES: W: CA, JP, US; RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE. (German). CODEN: PIXXD2. APPLICATION: WO 2000-EP7915 20000814. PRIORITY: DE 1999-19938704 19990814.
AB The invention relates to a compn. for implantation in the human and animal body as a **bone** substitute contg. inter alia calcium and phosphorous and comprises in particular mixts. of powders and base liqs. They are suitable for the prodn. of calcium phosphate-cement pastes which cure at room and/or body temp. The reaction systems contain a powdery base mixt. of $CaKPO_4$, $Ca_2NaK(PO_4)_2$ and $Ca(H_2PO_4)_2.H_2O$.
IT **7778-18-9**, Calcium sulfate **10101-41-4**, **Calcium sulfate dihydrate**
(**bone** substitute for implantation in the human and animal body)
RN 7778-18-9 HCA
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

RN 10101-41-4 HCA
CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



Ca

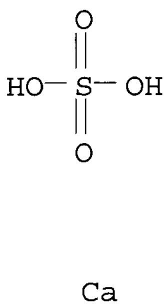
● 2 H_2O

IC ICM A61L024-00
ICS A61L027-10; A61L027-12; A61K006-033
CC 63-7 (Pharmaceuticals)
ST **bone** substitute implant calcium phosphate cement
IT Medical goods
(**bone** cements; **bone** substitute for implantation in the human and animal body)
IT Antibiotics
Disinfectants
(**bone** substitute for implantation in the human and animal body)
IT Amelogenins
Bone morphogenetic proteins
Growth factors, animal
(**bone** substitute for implantation in the human and animal body)
IT **Bone**
(implant; **bone** substitute for implantation in the human and animal body)

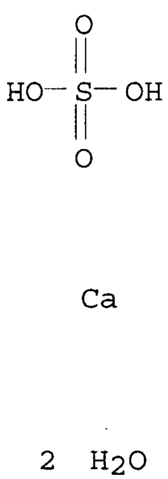
IT Pastes
(medical; **bone** substitute for implantation in the human and animal body)
IT Drug delivery systems
(nanoparticles; **bone** substitute for implantation in the human and animal body)
IT **Bone**
Tooth
(repair of; **bone** substitute for implantation in the human and animal body)
IT 471-34-1, Calcium carbonate, biological studies 7757-93-9, Calcium hydrogen phosphate **7778-18-9**, Calcium sulfate
10031-30-8, Calcium bis-dihydrogen phosphate monohydrate
10034-76-1, Calcium sulfate hemihydrate **10101-41-4**, **Calcium sulfate dihydrate** 15634-16-9
18901-69-4 131862-42-5
(**bone** substitute for implantation in the human and animal body)
IT 3672-15-9, Mannose 6 phosphate 57680-56-5, Sucrose octasulfate
57680-56-5D, Sucrose octasulfate, potassium and sodium complexes
(**bone** substitute for implantation in the human and animal body)
IT 9004-06-2, Elastase
(inhibitor; **bone** substitute for implantation in the human and animal body)
IT 1306-06-5, Hydroxylapatite
(nanoparticulate; **bone** substitute for implantation in the human and animal body)

L71 ANSWER 9 OF 23 HCA COPYRIGHT 2003 ACS on STN
132:352850 Calcium phosphate cements containing polyalkene acids. Wenz, Robert; Boltong, Trudy (Merck Patent G.m.b.H., Germany). Eur. Pat. Appl. EP 1002513 A1 20000524, 9 pp. DESIGNATED STATES: R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO. (German). CODEN: EPXXDW. APPLICATION: EP 1999-122195 19991106. PRIORITY: DE 1998-19853832 19981121.
AB Biocompatible, biodegradable Ca phosphate cements useful in dental cements, **bone** cements, **bone** substitutes, **bone** fillers, **bone** adhesives, etc. are provided which show a practical (not too rapid) setting time, low brittleness, high breaking strength, and good adhesion to **bone**. These cements are based on a solid phase of .alpha.- and/or .beta.-tri-Ca phosphate and/or CaSO4 mixed with a liq. phase contg. polyalkenoic acids , e.g. poly(meth)acrylic acid, or their salts or copolymers, esp. with malonic acid, itaconic acid, or allenes. The polyalkenoic acids have mol. wt. <8000 to facilitate their renal excretion. Thus, 25% aq. polyacrylic acid soln. was mixed 1:1 with.alpha.-tri-Ca phosphate (mean particle size 10 .mu.m). The mixt. began to set within 2 min 25 s at 20.degree., and hardening was complete within 6 min 30 s; the compression strength after immersion of the hardened cement in Ringer's soln. was 18 and 43 MPa after 1 and 4 days, resp.

IT **7778-18-9**, Calcium sulfate **10101-41-4**,
Calcium sulfate dihydrate
(**calcium** phosphate cements contg. polyalkene acids)
RN 7778-18-9 HCA
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



RN 10101-41-4 HCA
CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



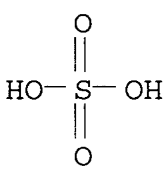
IC ICM A61K006-033
CC 63-7 (Pharmaceuticals)
ST cement **bone** dental calcium phosphate polyalkenoate; polyacrylate calcium phosphate **bone** dental cement
IT Adhesives
(biol., for **bone**; calcium phosphate cements contg. polyalkene acids)
IT Medical goods
(**bone** cements; calcium phosphate cements contg. polyalkene acids)
IT Fillers
(for **bone**; calcium phosphate cements contg. polyalkene acids)

IT **Bone**
(substitute; calcium phosphate cements contg. polyalkene acids)
IT 97-65-4D, Itaconic acid, polymers with alkenoic acids 141-82-2D, Malonic acid, polymers with alkenoic acids 471-34-1, Calcium carbonate, biological studies 1306-01-0, Calcium oxide phosphate (Ca4O(PO4)2) 1306-04-3, Chloroapatite 1306-06-5, Hydroxylapatite 7757-93-9, Calcium hydrogen phosphate (CaHPO4) **7778-18-9**, Calcium sulfate 7779-90-0, Zinc phosphate 7789-77-7 9003-01-4, Poly(acrylic acid) 10034-76-1, Calcium sulfate hemihydrate 10043-83-1 **10101-41-4**, **Calcium sulfate dihydrate** 12013-61-5, Calcium chloride phosphate (Ca2Cl(PO4)) 12167-74-7, Calcium hydroxide phosphate (Ca5(OH)(PO4)3) 13780-17-1, Calcium sodium phosphate (CaNaPO4) 15892-70-3 18901-69-4, Calcium potassium phosphate (CaKPO4) 21028-45-5 25549-84-2, Poly(sodium acrylate) 25948-33-8, Acrylic acid/itaconic acid copolymer 26159-89-7, Poly(potassium acrylate) 88938-16-3, Calcium sodium phosphate [Ca10Na(PO4)7] 131862-42-5 270080-66-5 270080-67-6
(**calcium** phosphate cements contg. polyalkene acids)

L71 ANSWER 10 OF 23 HCA COPYRIGHT 2003 ACS on STN
131:350772 Manufacture of coated fertilizer and its equipment. Chen, Dazhao; Yan, Zongbiao; Chen, Jihui; Yuan, Jiyan; He, Weijia; Chen, Suzhen (Guangzhou Nitrogenous Fertilizer Plant, Peop. Rep. China). Faming Zhuanli Shenqing Gongkai Shuomingshu CN 1150941 A 19970604, 33 pp. (Chinese). CODEN: CNXXEV. APPLICATION: CN 1996-105688 19960606.
AB The fertilizer is composed of granular fertilizer and coating material. The coating material is composed of animal glue 0.1-50, solubilizer 1-180, org. acid 0.05-30, inorg. acid 0.1-15, plant nutrient compd. 0.1-200, surfactant 0.1-15, and water 5-450 parts, preferably animal glue 1-10, solubilizer 10- 100, org. acid 1-10, inorg. acid 1-5, plant nutrient element compd. 10-120, surfactant 2-7, and water 100-300 parts. The org. acid is selected from one or more of maleic acid, oxalic acid, lauric acid, formic acid, acetic acid, humic acid, citric acid, and adipic acid; the inorg. acid from one or more of H3PO4, HCl, H2SO4, HNO3, and H3BO3; the animal glue from one or more of hide glue, **bone** glue, and mixed glue; the plant nutrient element compd. from one or more of sulfate of Fe, Zn, Mn, Mo, Cu, Mg, Ti, and K; the solubilizer from one or more of urea, (NH4)2SO4, NH4NO3, MgSO4, and NH4 citrate; and the surfactant from quaternary cationics. The granular fertilizer is selected from one or more chem. fertilizer of N fertilizer, P fertilizer, and K fertilizer, and humic acid fertilizer. The N fertilizer is selected from one or more of urea, NH4NO3, NH4Cl, (NH4)2SO4, and **Ca (NO3)2**; the P fertilizer from one or more of triple superphosphate, double superphosphate, (NH4)3PO4, ammonium nitrophosphate, ammonium ureido-phosphate (Niaolin'an), NH4 polyphosphate, nitrophoska, fused Ca-Mg phosphate, defluorinated phosphate, Thomas phosphate, ordinary superphosphate, calcium phosphate, Ca phosphate, and ground phosphate rock fertilizer; and the humic acid fertilizer from one or more of Na humate, K humate,

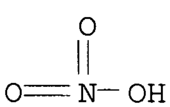
NH4 humate, humic acid, nitro-humic acid. The coated fertilizer is prepd. by inhaling coating material in sprayer by 0.3-0.5 MPa compressed air, coating the granular by spraying coating material, **drying**, and oxidizing. The addn. of the coating material is 6-10 kg/ton. The equipment is composed of reactor, filter, pump, storage tank, elevated tank, coating sprayer, and pelleting tower. The coating sprayer is composed of air-spraying pipe, coat-emulsifying pipe, const.-diam. spray pipe, nozzle, and air entrance. The const.-diam. spray pipe is connected with coat-emulsifying pipe at one mouth, with nozzle at another mouth. The coat-emulsifying pipe is composed of a flange at its front-end, a content-diam. piper at the medium, a columnar cavity connected with two expanded-diam. pipes at each end between flange and medium, and a coat-entrance pipe mounted nearby flange. The spout diam. of nozzle is 6-8 mm.

IT **7783-20-2**, Ammonium sulfate, biological studies
 10124-37-5, Calcium nitrate
 (manuf. of coated fertilizer and its equipment)
RN 7783-20-2 HCA
CN Sulfuric acid diammonium salt (8CI, 9CI) (CA INDEX NAME)



●2 NH_3

RN 10124-37-5 HCA
CN Nitric acid, calcium salt (8CI, 9CI) (CA INDEX NAME)



1/2 Ca

IC ICM C05G003-00
 ICS C05G005-00
CC 19-6 (Fertilizers, Soils, and Plant Nutrition)
IT 57-13-6, Urea, biological studies 64-18-6, Formic acid, biological studies 64-19-7, Acetic acid, biological studies 77-92-9, Citric acid, biological studies 110-16-7, Maleic acid, biological studies

124-04-9, Adipic acid, biological studies 143-07-7, Lauric acid, biological studies 144-62-7, Oxalic acid, biological studies 1314-62-1, Vanadium oxide, biological studies 6484-52-2, Ammonium nitrate, biological studies 7487-88-9, Magnesium sulfate, biological studies 7631-95-0, Sodium molybdate 7632-50-0, Ammonium citrate 7664-38-2, Phosphoric acid, biological studies 7664-93-9, Sulfuric acid, biological studies 7697-37-2, Nitric acid, biological studies 7720-78-7, Ferrous sulfate 7733-02-0, Zinc sulfate **7783-20-2**, Ammonium sulfate, biological studies 7785-87-7, Manganese sulfate 7790-93-4, Chloric acid 10043-35-3, Boric acid, biological studies 10103-46-5, Calcium phosphate **10124-37-5**, Calcium nitrate 11098-84-3, Ammonium molybdate 12125-02-9, Ammonium chloride, biological studies 13463-67-7, Titanium oxide, biological studies
(manuf. of coated fertilizer and its equipment)

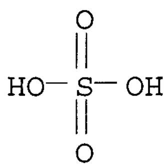
L71 ANSWER 11 OF 23 HCA COPYRIGHT 2003 ACS on STN
124:15549 Calcium phosphate calcium sulfate composite implant material. Liu, Sung Tsuen; Chung, Harvey H. (USA). U.S. US 5462722 A 19951031, 5 pp. (English). CODEN: USXXAM. APPLICATION: US 1991-687592 19910417.

AB A new inorg. composite materials for hard tissue replacement composite material comprises solid material of calcium sulfate which is fully or partially converted to calcium phosphate from aq. soln. This composite material has good biocompatibility and controllable resorption, and will be very useful for **bone** substitute material in orthopedic and dental applications. The fully converted material which comprises mainly apatite calcium phosphate is also useful for chromatog. application. Calcium sulfate hemihydrate, particle size of 20-60 mesh 5 g was added to a 11% soln. of diammonium hydrogen phosphate, pH = 10, and heated at 80.degree. for 12 h during which time period the starting material was converted to a calcium phosphate material. After that, the suspension was filtered and sepd. solid particles were washed with water and dried. Anal. showed that the particles retained their original cryst. shape and the final solid dissolved more readily than the original **calcium sulfate dihydrate**, indicating substantially full conversion of **calcium sulfate dihydrate** to **calcium** phosphate during the heating process.

IT **7778-18-9**, Calcium sulfate **10101-41-4**, **Calcium sulfate dihydrate**
(**calcium** phosphate **calcium sulfate** composite implant material)

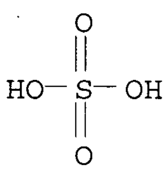
RN 7778-18-9 HCA

CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

RN 10101-41-4 HCA
CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



Ca

2 H_2O

IC ICM C01B015-16
ICS C01B025-26; C01F011-46; A61F002-28
NCL 423311000
CC 63-7 (Pharmaceuticals)
Section cross-reference(s): 78
IT 1310-58-3, Potassium hydroxide, reactions 1310-73-2, Sodium hydroxide, reactions 1336-21-6, Ammonium hydroxide 7558-79-4, Disodium hydrogen phosphate 7632-05-5, Sodium phosphate 7664-38-2, Phosphoric acid, reactions **7778-18-9**, Calcium sulfate 7783-28-0 10034-76-1, Calcium sulfate hemihydrate **10101-41-4**, **Calcium sulfate dihydrate** 10377-52-3, Lithium phosphate 13765-96-3 16068-46-5, Potassium phosphate
(**calcium** phosphate **calcium sulfate** composite implant material)

L71 ANSWER 12 OF 23 HCA COPYRIGHT 2003 ACS on STN
120:144256 Resorbable surgical cements. Liu, Sung Tsuen (USA). U.S. US

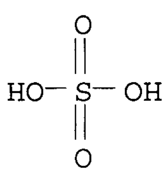
5281265 A 19940125, 5 pp. (English). CODEN: USXXAM. APPLICATION: US 1992-830381 19920203.

AB A surgical cement for use in medical applications such as orthopedic and maxillofacial surgeries and dental applications, comprises a hardened cement formed from a mixt. contg. (1) Ca components having a water soly. at 25.degree. (0.5-20).times.10-2M, (2) a setting component selected from the group consisting of water-sol. salts of polyfunctional C2-10-carboxylic acids, water-sol. dibasic phosphate salts, and mixts. thereof, and (3) water in an amt. effective to form a paste from the mixt. For example, a mixed powder of 2g **CaSO4**.cntdot.**2H2O** and 0.5g K citrate was stirred with 0.6 mL deionized water to give a viscous and cohesive paste. The paste became hardened and set for .apprx.2 mins. After aging in a water environment, this setting cement maintained its integrity and did not disintegrate.

IT **7778-18-9**, Calcium sulfate **10101-41-4**, **Calcium sulfate dihydrate**
(surgical cements contg. setting agents and, resorbable)

RN 7778-18-9 HCA

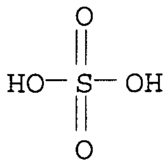
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

RN 10101-41-4 HCA

CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)

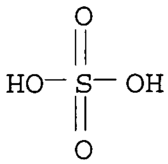


● Ca

2 H_2O

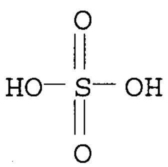
IC ICM C09K003-00
NCL 106035000
CC 63-7 (Pharmaceuticals)
IT Medical goods
(**bone** cements, calcium compds. and salts as setting agents in)
IT Animal growth regulators
(**bone**-derived growth factors, surgical cements contg.)
IT 140-99-8, Calcium succinate **7778-18-9**, Calcium sulfate 10034-76-1, Calcium sulfate hemihydrate **10101-41-4**, **Calcium sulfate dihydrate** 17482-42-7, Calcium malate 19455-76-6, Calcium malonate 34938-90-4, Calcium maleate
(surgical cements contg. setting agents and, resorbable)

L71 ANSWER 13 OF 23 HCA COPYRIGHT 2003 ACS on STN
120:38217 Resorbable bioactive phosphate containing cements. Liu, Sung Tsuen; Chung, Harvey H. (USA). U.S. US 5262166 A 19931116, 5 pp. (English). CODEN: USXXAM. APPLICATION: US 1991-687586 19910417.
AB A surgical cement of high biocompatibility, useful in orthopedic, maxillofacial, and dental applications comprises a Ca alkali phosphate and a citrate. Thus, stoichiometric amts. of CaHPO4 and Na2CO3 were mixed and sintered at high temp. to form CaNaPO4 ceramic (I). I powder 2, was mixed with citric acid 8 g and a few drops of water was then added to form a sticky paste which was set after few mins. The surface pH of the set cement was .gtoreq.7 and hardened cement aged in pure water did not show any sign of disintegration.
IT **7778-18-9**, Calcium sulfate **10101-41-4**, **Calcium sulfate dihydrate**
(resorbable bioactive phosphate cements contg. calcium alkali phosphates and citrates and)
RN 7778-18-9 HCA
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

RN 10101-41-4 HCA
CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



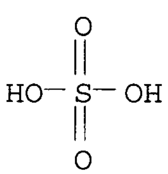
Ca

2 H_2O

IC ICM A61F002-02
ICS A61F002-28; C09K003-10
NCL 424423000
CC 63-7 (Pharmaceuticals)
Section cross-reference(s): 78
IT Medical goods
(**bone** cements, prepn. of, from calcium alkali phosphate and citrate)
IT Animal growth regulators
(**bone**-derived growth factors, resorbable bioactive phosphate cements contg. calcium alkali phosphates and citrates and)
IT 471-34-1, Calcium carbonate, biological studies 1305-62-0, Calcium hydroxide, biological studies 1305-78-8, Calcium oxide, biological studies 1309-42-8, Magnesium hydroxide 1309-48-4, Magnesium oxide, biological studies 7693-13-2, Calcium citrate 7757-93-9, Dicalcium phosphate 7758-87-4, .alpha.-Tricalcium phosphate **7778-18-9**, Calcium sulfate 7789-75-5, Calcium fluoride, biological studies **10101-41-4**, **Calcium**

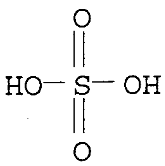
sulfate dihydrate 13767-12-9, Octacalcium phosphate
(resorbable bioactive phosphate cements contg. calcium alkali phosphates and citrates and)

L71 ANSWER 14 OF 23 HCA
117:239897 Resorbable bioactive calcium phosphate cement. Liu, Sung Tsuen; Chung, Harvey H. (USA). U.S. US 5149368 A 19920922, 5 pp. (English). CODEN: USXXAM. APPLICATION: US 1991-639536 19910110.
AB A cement paste for orthopedic, dental, and maxillofacial applications is prepd. from a (2-15):1 mixt. of a tetracalcium phosphate cementing powder, a setting agent, and water, the paste having pH >5 after the setting. The setting agent is selected from NaH2 citrate, Na2H citrate, KH2 citrate, K2H citrate, (NH4)2H citrate, and NH4H2 citrate. The paste may further contain an inert filler, e.g., .alpha.-tricalcium phosphate.
IT **7778-18-9 10101-41-4, Calcium sulfate dihydrate**
(dental and **bone** cement contg.)
RN 7778-18-9 HCA
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

RN 10101-41-4 HCA
CN Sulfuric acid, calcium salt (1:1), dihydrate (8CI, 9CI) (CA INDEX NAME)



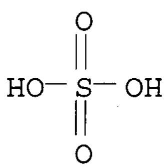
Ca

2 H_2O

IC ICM C09K003-00
NCL 424602000
CC 63-7 (Pharmaceuticals)
IT 1306-01-0, Tetracalcium phosphate
(cement paste contg., bioactive, for **bone** and tooth)
IT 144-33-2, Disodium hydrogen citrate 471-34-1, Calcium carbonate, biological studies 866-83-1, Potassium dihydrogen citrate 1305-62-0, Calcium hydroxide, biological studies 1305-78-8, Calcium oxide, biological studies 1309-42-8, Magnesium hydroxide 1309-48-4, Magnesium oxide, biological studies 3012-65-5, Diammonium hydrogen citrate 3609-96-9, Dipotassium hydrogen citrate 4450-94-6, Ammonium dihydrogen citrate 7693-13-2, Calcium citrate 7757-93-9, Dicalcium phosphate 7758-87-4, .alpha.-Tricalcium phosphate **7778-18-9** 7789-75-5, Calcium fluoride, biological studies 10034-76-1, Calcium sulfate hemihydrate **10101-41-4, Calcium sulfate dihydrate** 10103-46-5 13767-12-9, Octacalcium phosphate 18996-35-5, Sodium dihydrogen citrate
(dental and **bone** cement contg.)
IT 1310-73-2, Sodium hydroxide, biological studies
(in prepn. of dental and **bone** cement)

L71 ANSWER 15 OF 23 HCA
103:200899 Implant materials containing ceramics and solution of alginate and phosphate salt in two-compartment packet. De Groot, Klaas (Neth.). Neth. Appl. NL 8304129 A 19850701, 8 pp. (Dutch). CODEN: NAXXAN. APPLICATION: NL 1983-4129 19831201.
AB A paste contg. a bioactive ceramic (particle size .gtoreq.200 .mu.m), CaSO4, and an aq. soln. of alginate and phosphate salts is useful for filling cavities in teeth and **bones**. Formation of a Ca alginate gel during hardening of the paste is modulated by the phosphate. The paste is formed from a powd. and a liq. component, stored sep. in a 2-compartment packet. For example, a powd. mixt. of K alginate [9005-36-1] 6, CaCO3 granules (particle

size 250-350 .mu.m) 90, and **CaSO4.2H2O** 4 g was
mixed with 40 g 0.2% aq. K phosphate soln. to form a paste.
IT **7778-18-9**
(**bone** cement and dental filling contg.)
RN 7778-18-9 HCA
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



Ca

IC ICM A61L027-00
ICS A61K006-06; C04B035-66
CC 63-7 (Pharmaceuticals)
ST filling tooth **bone** calcium alginate
IT Apatite-group minerals
(**bone** cement and dental filling contg. alginate and phosphate and calcium sulfate and)
IT Surgical dressings and goods
(**bone** cements, alginate and phosphate and calcium sulfate in)
IT **7778-18-9** 9005-36-1 16068-46-5
(**bone** cement and dental filling contg.)
IT 471-34-1, biological studies 14358-97-5
(**bone** cement and dental filling contg. alginate and phosphate and calcium sulfate and)

L71 ANSWER 16 OF 23 HCA COPYRIGHT 2003 ACS on STN
102:66394 Influence of CaSO4.cntdot.2H2O, CaSO4.cntdot.1/**2H2O** and **CaSO4** on the **initial hydration** of clinker having different burning degree. Uchikawa, Hiroshi; Uchida, Shunichiro; Ogawa, Kenji; Hanehara, Shunsuke (Cent. Res. Lab., Onoda Cement Co., Ltd., Tokyo, Japan). Cement and Concrete Research, 14(5), 645-56 (English) 1984. CODEN: CCNRAI. ISSN: 0008-8846.
AB Relations between early **hydration** process and the properties of fresh cement until setting was studied by using 9 kinds of cements prepd. from 3 com. clinkers with various burning degree and kinds of Ca sulfates, i.e., natural gypsum, .beta.-hemihydrate, and anhydrite II. The Ca(OH)2 and CaSO4 satn. ratio in the liq. phase had a marked effect on the early **hydration** process of cement paste rather than the hydraulic reactivity of clinker minerals themselves. The setting time corresponded well to the period of max. Ca(OH)2 satn. ratio in the liq. phase which also coincided with the period of vivid

hydration of C3S. Soft burning of clinker so as to leave a small amt. (0.5% level) of free lime was effective for shortening the setting time but the long-term strength was slightly inferior to that of cement from well burnt clinker. Through the setting time was shortened in the order: natural gypsum, .beta.-hemihydrate, and anhydrite II in cement from well burnt clinker, the kind of Ca sulfate had no marked effect on cement from poorly burnt clinker.

CC 58-1 (Cement, Concrete, and Related Building Materials)

ST calcium sulfate cement **hydration**

IT Cement
(**hydration** of, effect of clinker burning degree and calcium sulfate type on)

IT 13397-24-5, uses and miscellaneous 14798-04-0 26499-65-0
(in **hydration**, of cement, clinker burning degree in relation to)

L71 ANSWER 17 OF 23 HCA COPYRIGHT 2003 ACS on STN

101:78801 Effect of sodium citrate on expansion reversal of gypsum-bonded investments. Fukui, Hisao; Kito, Masashi; Hisada, Kazuaki; Kitaoka, Tadashi; Hasegawa, Jiro (Sch. Dent., Aichi-Gakuin Univ., Nagoya, 464, Japan). Shika Zairyo, Kikai, 3(2), 303-9 (Japanese) 1984. CODEN: SZKIDA. ISSN: 0286-5858.

AB Combined effects of admixed Na citrate [68-04-2] and CaSO4.cntdot.2H2O on the setting expansion of a investment which **initially** contains no **CaSO4**.cntdot.**2H2O** were studied. Na citrate retarded the initial setting time for expansion, retarded the time to achieve max. expansion, and reduced or eliminated the loss of setting expansion. When samples were mixed with 0.05% Na citrate soln., the **crystal** of CaSO4.cntdot.2H2O and porosity of set investment grew larger compared with mixed water. There was a strong correlation between the loss of setting expansion and the rate of set of the investment.

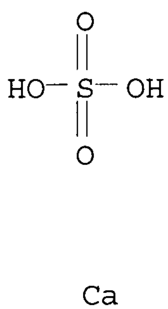
CC 63-7 (Pharmaceuticals)

L71 ANSWER 18 OF 23 HCA COPYRIGHT 2003 ACS on STN

97:81282 Computation and measurement of absorbed dose in **bone** due to .beta.-particles. Chen, Huan Tong; Chang, Pao Shu; Weng, Pao Shan (Health Phys. Div., Inst. Nucl. Energy Res., Lung-Tan, Taiwan). Hoken Butsuri, 16(4), 291-6 (English) 1981. CODEN: HOKBAQ. ISSN: 0367-6110.

AB A thermoluminescent (CaSo4:Dy) dosimeter (TLD) for measurement of .beta.-particle from 90Sr in femur was studied. The TLD powder (100-300 .mu.m in particle size) was prepd. by mixing 75 mg of Dy2O3 in 300 mL H2SO4 and 34.4 g of **CaSO4.2H2O**, evapg. at 300.degree., and pulverizing by an automatic dispenser. A 5.0 mg of the powder was sealed in a polyethylene capsule 1.65 mm in outside diam. and 9.0 mm in length, and was calibrated in a series of std. solns. of 90Sr-90Y. The capsule and a std. .beta.-source were also inserted in 2 yellow-marrow cavities which are sepd. by a known distances. Comparison between the results and a simulation model with the Monte Carlo technique are given, and some sources of errors were also discussed.

IT **7778-18-9**
(thermoluminescent dosimeters of, dysprosium doping with dysprosium oxide in)
RN 7778-18-9 HCA
CN Sulfuric acid, calcium salt (1:1) (8CI, 9CI) (CA INDEX NAME)



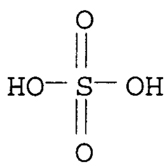
CC 71-7 (Nuclear Technology)
Section cross-reference(s): 8
IT **Bone**
(thermoluminescent dosimeter for .beta.-particle from)
IT Dosimeters
(thermoluminescent, dysprosium-doped calcium sulfate for .beta.-particle from **bone**)
IT **7778-18-9**
(thermoluminescent dosimeters of, dysprosium doping with dysprosium oxide in)

L71 ANSWER 19 OF 23 HCA COPYRIGHT 2003 ACS on STN
96:82194 Quantitative EPMA of biological tissue using mixtures of salts as standards. Krefting, E. R.; Lissner, G.; Hoehling, H. J. (Inst. Med. Phys., Univ. Muenster, Muenster, D-4400, Fed. Rep. Ger.). Scanning Electron Microscopy (2), 369-76 (English) 1981. CODEN: SEMYBL. ISSN: 0586-5581.
AB Freeze-**dried**, unembedded cryostat sections of shock-frozen tissues were analyzed by electron probe microanal. (EPMA), and the equation of T. A. Hale (1971) was applied to obtain quant. concns. of elements. The stds. currently available seem to be of limited accuracy (e.g. stds. of macrocyclic complex salts, embedded in Spurr's epoxy resin, are unstable under the electron beam and often inhomogeneous). Therefore, stds. were prepd. as follows: mixts. of inorg. salts were dissolved in distd. water, pipetted in thin lines onto the supporting film, rapidly frozen in liq. N, and finally freeze **dried**. These stds. are thin and stable under the electron beam; even mixts. of salts are homogeneous in compn. when analyzing areas of 10 .times. 10 .mu.m2. By using different salts, differences in compn. between std. and specimen did not influence the quant. results. With these stds., element concns. were measured in the epiphyseal growth plate. The P content is intracellularly very high (.apprx.4 wt.%/**dry** mass) but extracellularly relatively low (.apprx.0.2%). This is valid in all zones of the

unmineralized growth plate. However, the mean values are different because the nos. of cells per vol. differ in the different cell zones.

IT **7778-80-5**, uses and miscellaneous **10043-52-4**, uses and miscellaneous
(std. contg., for electron microprobe anal. of biol. materials)

RN 7778-80-5 HCA
CN Sulfuric acid dipotassium salt (8CI, 9CI) (CA INDEX NAME)



2 K

RN 10043-52-4 HCA
CN Calcium chloride (CaCl2) (9CI) (CA INDEX NAME)

Cl— Ca— Cl

CC 9-5 (Biochemical Methods)
IT **Bone**, composition
(epiphyseal plate, elements detn. in, by electron microprobe anal., stds. for)

IT 7447-40-7, uses and miscellaneous 7647-14-5, uses and miscellaneous 7778-77-0 **7778-80-5**, uses and miscellaneous 7786-30-3, uses and miscellaneous **10043-52-4**, uses and miscellaneous
(std. contg., for electron microprobe anal. of biol. materials)

L71 ANSWER 20 OF 23 HCA COPYRIGHT 2003 ACS on STN
65:77782 Original Reference No. 65:14533g-h Modification of the dihydrate of calcium sulfate. Berg, L. G.; Spiridonov, F. P.; Zdanovskii, A. B. (State Univ., Kazan). Doklady Akademii Nauk SSSR, 169(3), 583-6 (Russian) 1966. CODEN: DANKAS. ISSN: 0002-3264.

AB A **new**, metastable modification of **CaSO4.2H2O** (I), called .alpha.-gypsum (II) forms on dissolving of very clean .alpha.-CaSO4.0.5H2O (no admixts. of .beta.-CaSO4.0.5H2O and normal .beta.-I) in H2O. II changes slowly to normal .beta.-I, whereby this process is accelerated by addn. of .beta.-I **crystals**. The soly. of II is higher than that of the normal .beta. modification and its thermographic investigation gives a different heating curve. The II **crystals** are needle-shaped with a characteristic oblique extinction with .gamma. = 1.523 and .alpha. = 1.510, compared to .gamma. = 1.530 and .alpha.

= 1.520 for normal gypsum.
CC 8 (Crystallization and Crystal Structure)

L71 ANSWER 21 OF 23 HCA COPYRIGHT 2003 ACS on STN
47:10623 Original Reference No. 47:1912f-g Effects of agents added on the setting expansion of plaster of Paris. Ito, Shuyo; Teraoka, Ichiro; Yoshihama, Seiichiro (Shibaura Elec. Co., Tokyo). Sekko, 1, 282-7 (Unavailable) 1952. CODEN: SEKKA2. ISSN: 0370-954X.
AB The setting expansion (generally with **initial** contraction) of .beta.-**CaSO4**.1/**2H2O** at room temp. with stirring after adding, either alone or in combination, inorg. (cement, bentonite, active clay, fuller's earth, Rochelle salt, KBr, and NH4NO3) or org. (sucrose and Na citrate) compds. with (const. or varying amts.) or without H2O was detd. to study the mechanism of **hydration**. The expansion was influenced by the soly., and H2O in excess resulted in smaller expansion (or contraction).
CC 20 (Cement, Concrete, and Other Building Materials)
IT **Hydration** (chemical)
(of plaster of Paris, additive effect on)

L71 ANSWER 22 OF 23 HCA COPYRIGHT 2003 ACS on STN
26:34588 Original Reference No. 26:3604c-f Solubility of calcium phosphates in solutions of the salts used as fertilizers. Lenglen; Milhiet Chimie et Industrie (Paris), Special No., 823-5 (Unavailable) 1932. CODEN: CHIEAN. ISSN: 0009-4358.
AB A report is given of the results obtained to date in the course of an as yet uncompleted investigation. Salts can be placed in the following decreasing order: for acid radicals-sulfates, chlorides, nitrates; for bases-NH4, Mg, Na, K. Com. (NH4)2SO4 has a higher solubilizing action than the C. P. salt. Mixts. of salts have a slightly lower effect than the sum of the actions of the individual salts. Water satd. with CO2 (1.8 g. per l.) exerts a much stronger action than any of the salts studied; if the salt solns. are satd. with CO2, the actions of the CO2 and of the salt are additive; if the CO2 soln. is satd. with CaCO3 its action is considerably reduced. **CaCl2** exerts no solubilizing action, and Na2CO3 and K2CO3 exert but little. Fe sulfate and Fe NH4 sulfate act in the same way as **Na2SO4** and **K2SO4**; CuSO4 is more active. Al2(SO4)3 solubilizes a considerable proportion of P2O5, but this seems to be due to the free acidity produced by hydrolysis. The solubilizing action increases with the concn. of the salt soln., but at a rate which decreases with increase in concn. On decreasing the amt. of phosphate brought into contact with the salt soln., the proportion of P2O5 dissolved at first increases fairly rapidly and then decreases. Solubilization increases fairly rapidly with the time of agitation. Gelatinous Ca3(PO4)2 prepd. in the lab. is more readily dissolved than the same after **drying** at 110.degree.. CaHPO4 prepd. from **bones** dissolves about twice as readily as CaHPO4 prepd. from mineral phosphates.
CC 15 (Soils, Fertilizers, and Agricultural Poisons)

L71 ANSWER 23 OF 23 HCA COPYRIGHT 2003 ACS on STN

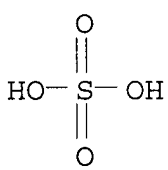
16:3117 Original Reference No. 16:603i,604a-b Soil investigations.
Anon. Calif. Sta. Report pp. 59-65 (Unavailable) 1919.

AB Californian N-poor soils have an abnormal capacity for nitrification. Sol. salts liberate Ca and become insol. Cl, SO4, and NO3 are but slightly absorbed. The soil acidity is due to free acid (H-ion). The relative order of availability of N in nitrogenous fertilizers is: (NH4)2SO4, NaNO3, **Ca(NO3)2**, **dried** blood, tankage, steamed **bone** meal. S when applied with N fertilizers increases the availability of the N. The soil in good spots of poor grain fields was 6 to 10 times as powerful in producing NO3 as the soil outside of the good spots. Peat is useless as a source of N on N-poor soils. Gypsum and lime increase the sol. K in some soils and not in others. The ratio of water sol. ext. in cropped soils to concurrent draft was large. Fresh manure causes a temporary reduction of NO3 in the soil. Banana stalks and tule contain quantities of K.

IT **7783-20-2**, Ammonium sulfate **10124-37-5**, Calcium nitrate
(nitrogen availability in)

RN 7783-20-2 HCA

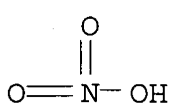
CN Sulfuric acid diammonium salt (8CI, 9CI) (CA INDEX NAME)



2 NH_3

RN 10124-37-5 HCA

CN Nitric acid, calcium salt (8CI, 9CI) (CA INDEX NAME)



1/2 Ca

CC 15 (Soils, Fertilizers, and Agricultural Poisons)

IT Blood
(**dried**, nitrogen availability in)

IT **Bones**
(meal, nitrogen availability in)

IT 7631-99-4, Sodium nitrate **7783-20-2**, Ammonium sulfate
10124-37-5, Calcium nitrate
(nitrogen availability in)